LABORATORY GAS CHROMATOGRAPHS

214.2.840.039UM "Chromatec-Crystal 5000"

214.2.840.075UM "Chromatec-Crystal 9000"

USER MANUAL

PART 2

INLETS, GAS VALVES, DETECTORS

Edition 05.09.2023

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Attention! ..... **Ошибка! Закладка не определена.**

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INTRODUCTION

Contamination of columns or detectors is not covered by the warranty.

All the maintenance procedures of the chromatograph components (inlets, valves, detectors, etc.) described below are not complicated and are carried out by the operator when working with the chromatograph.

Instructions marked by this symbol are necessary for carrying out in order to avoid injuries sustaining at working with chromatograph.



Instructions marked by this symbol are to be carried out when there is electron-capture detector (ECD) in chromatograph, which containing radioactive source Ni^{63}.



This symbol makes warning about danger of burn. It is painted on:

Top cover of the gas chromatograph (hereinafter the GC) (warning about hot surfaces of injectors and detectors).

Rear cover of the GC indicating: "**Hot air**" (warning about danger of burn at oven heating and cooling cycles.



Instruction marked by this symbol is warning about explosion risk.

There is a sign in the oven door:

Attention! Before GC turning on make sure that hydrogen lines are sealed or connected to the column.

1 INLETS

Configuration of inlets and carrier gases is performed in accordance by section 3.2.1 Sample injection ports, User Manual 214.00045-51I, Chromatec Analytic software.

Setting the parameters of the inlet and feed gases in the analysis mode is performed in accordance by section 3.4.1 Sample injection ports, User Manual 214.00045-51I, Chromatec Analytic software.

1.1 Inlet types

Inlet is intended for injection of liquid and gaseous samples into chromatographic column using syringe. Many peripheral samplers (external devices for sample preparation and injection, such as thermal desorber, headspace sampler, liquefied gas sampler) are connected to “Chromatec-Crystal” gas chromatographs (hereinafter – the GC) via inlets.

Table 1.1 – Methods of sample injections

Column type	**Mode**	**Inlet type**
Packed	Splitless	Packed inlet 7.2
	On-column	
Capillary	Split	Capillary Inlet 7.2
	Splitless	
	Pressure impulse	
Capillary	Programmable with column oven heating rate split/splitless	Capillary Inlet 7.3 Programmable
	On-column	
	Pulsed pressure mode	
Capillary	On-column	On-column inlet7.4
Capillary	High-speed split/splitless	Programmable inlet 8.1
	Large volume injection	
	liquid-cooled cold injection	

Packed and capillary inlets 7.2 have the same body, and differ by intention in connected components and pneumatic control. Therefore, capillary inlet can be re-configured into packed inlet and vice versa.

Table 1.1 – Application for packed inlet 7.2

Column type	Mode	Application
Packed		Total volume injected into column. Packing can be placed in the liner to minimize "contamination" of the column with unwanted components.
		Injection entire sample volume into the column with elongated front end. Reduction of smearing of chromatographic peaks, is applied at some applications.
Capillary	Split	Split ratio reduces sample volume entered into column to avoid its overloading. Split mode is the best for thermostable analytes at level above 50 ppm.
	Splitless	Most part of the sample transferred into column at closed vent valve, after entering analytes, vent got opened and liner blown out. Splitless mode is convenient for low concentration, below 50 ppm. Analytes should be heavier than solvent.
		Sample injection by a pressure pulse is a more efficient transfer of the sample from the liner to the column. After sample injection the inlet pressure is restored to its normal value.

Table 1.2 – Application for Inlet 7.3 Programmable

Column type	Mode	Application
Capillary	Split	Split ratio reduces sample volume entered into column to avoid its overloading. Split mode is the best for thermostable analytes at level above 50 ppm.
	Splitless	Most part of the sample transferred into column at closed vent valve, after entering analytes, vent got opened and liner blown out. Splitless mode is convenient for low concentration, below 50 ppm. Analytes should be heavier than solvent.
		Sample injection by a pressure pulse is a more efficient transfer of the sample from the liner to the column. After sample injection the inlet pressure is restored to its normal value.
	On-column	Sample is injected directly into cooled column capillary, which front end is located in the inlet, then the inlet and the column are heated by program. On-column mode is recommended for determination of analytes in wide boiling point range. Sample fractioning is excluded comparatively to split or splitless mode.

Table 1.4 – Application for Inlet 7.4 On-column

Column type	Mode	Application
Capillary	On-column	Sample is injected directly into cooled column capillary, which front end is located in the inlet, then the inlet and the column are heated by program. On-column mode is recommended for determination of analytes in wide boiling point range. Sample fractioning is excluded comparatively to split or splitless mode.

Table 1.5 – Application for Inlet 8.1 Programmable

Column type	Mode	Application
Packed		Same as Inlet 7.2
Capillary	Split	Same as Inlet 7.2
	Splitless	Same as Inlet 7.2
	Large volume injection	Great sample volume (5 – 100 µl) is steadily injected into cooled inlet, solvent is blown out while analytes are focused in liner. Then vent valve shuts off, inlet temperature is heated quickly, target components are transferred into column. Thanks to greater sample volume, dramatically higher sensitivity can be obtained (10 – 50 times higher than in splitless mode).
	Cold injection	Cooling by liquid refrigerant: carbon dioxide or nitrogen

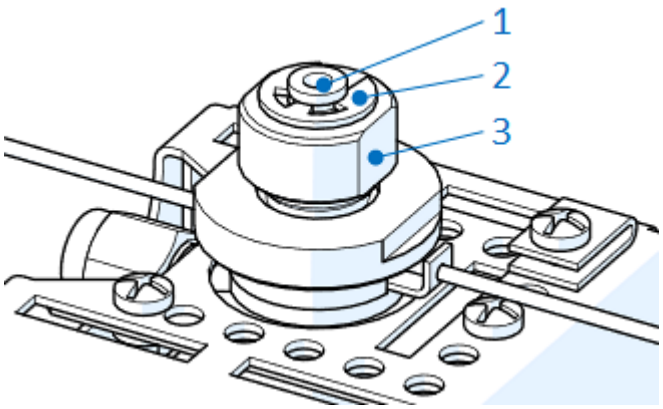
1.2 General recommendations

1.2.1 Needle guide usage

To minimize excessive tearing of the septum and ingress of septum particles into liner, internal diameter of needle guide must just slightly exceed needle diameter.

There several needle guide modifications for all types of inlets (Insert 8.220.184-XX) to be inserted into septum nut. Insert 8.220.184 (0.7mm hole) is located on default. Depending on the needle outer diameter suitable insert modifications are recommended (Table 1.). To replace insert the following procedure is recommended:

- unscrew the septum nut;
- remove the locking washer;
- change needle guide;
- do fit the locking washer back and screw the septum nut.



1 – Guide of needle, 2 – locking washer 3 – septum nut

Figure 1.1 – Needle guide in the septum nut

Table 1.6 – Needle guide modifications

Needle guide modification		**Syringes or needle gauge of syringes and samplers**	**Needle diameter, mm**
Description, Marking	Hole diameter, mm		
Insert 8.220.184	0.,7	Needle gauge 26	0.47
		Dual 23/26 needle gauge	0.64
Insert 8.220.184–01	1.1	Needle gauge 23	0.72
		TDS-1 (2-stage) thermal desorber	1.0

Depending on the model of autosampler, various types of inlet nuts are supplied in the autosamplers shipping kits.

1.2.2 Liner sealing

When using inlet at temperatures above 350 °C (as well as operating with thermal washer 13, Figure 1.2, Figure 1.5) rubber O-ring 11 can be worn out quite quickly and requires replacement often. O-ring wearing appears as residual deformation, sticking to inlet body surface and material properties deterioration (elasticity loss, fragility).

1.2.3 Septum usage

Inlet septum 9 (Figure 1.2, Figure 1.5) is a rubbery tablet-shaped having 10 or 11mm in diameter and 3 or 4 mm in thickness. Instead of standard septa delivered by Chromatec using low-bleed elastic septa is recommended.

Septum is sealed when screwing nut 4. Recommendations for the use and replacement of the inlet septum are given in section 1.7.2.

When septum 11mm in diameter is used, periodicity of screwing may be more seldom (after each 150 injections), this allows increasing penetrations quantity without leaks.

Take into account that hot inlet being leak free (at temperature 150° or higher) may disclose leak thorough the septum at lower temperatures, due to specific rubber properties. This situation is especially critical when working with the MSD detector, it is recommended to use septa with a diameter of 11 mm to eliminate the observed effect.

As soon as inlet temperature cooled down, check septum for leaks and tight septum nut slightly if necessary to avoid leaks through the septum.

When using inlet at temperature higher than 350°C (and especially with thermal washer 13), septum is exposed to be worn out, and requires to be timely changed. Besides with increased temperature of top part of inlet, the amount of volatile destruction products from septum may be so much that blowing channel is not able to remove it effectively. It may bring to getting destruction products to the column and ghost peaks on chromatogram. To avoid such effect at operating at temperatures above 350 °C and in all cases when using thermal washer special high-temperature septa are recommended to use (for example, HT SGE septa).

1.3 Packed Inlet 7.2

1.3.1 Packed inlet design

The inlet body (Figure 1.2), with a heater installed on it, is connected with the power casing 6 by the upper part.

The power casing allows you to orient the inlet body relative to the vertical axis. The design of the power casing is the same for all versions of inlets.

Inlet body has vent fitting 12, to which split vent line may be connected if packed inlet is inverted into split/splitless inlet.

Fitting sealed with cap for packed inlet. The bottom of the body has fitting 16 is screwed onto the inlet body to connect the packed column.

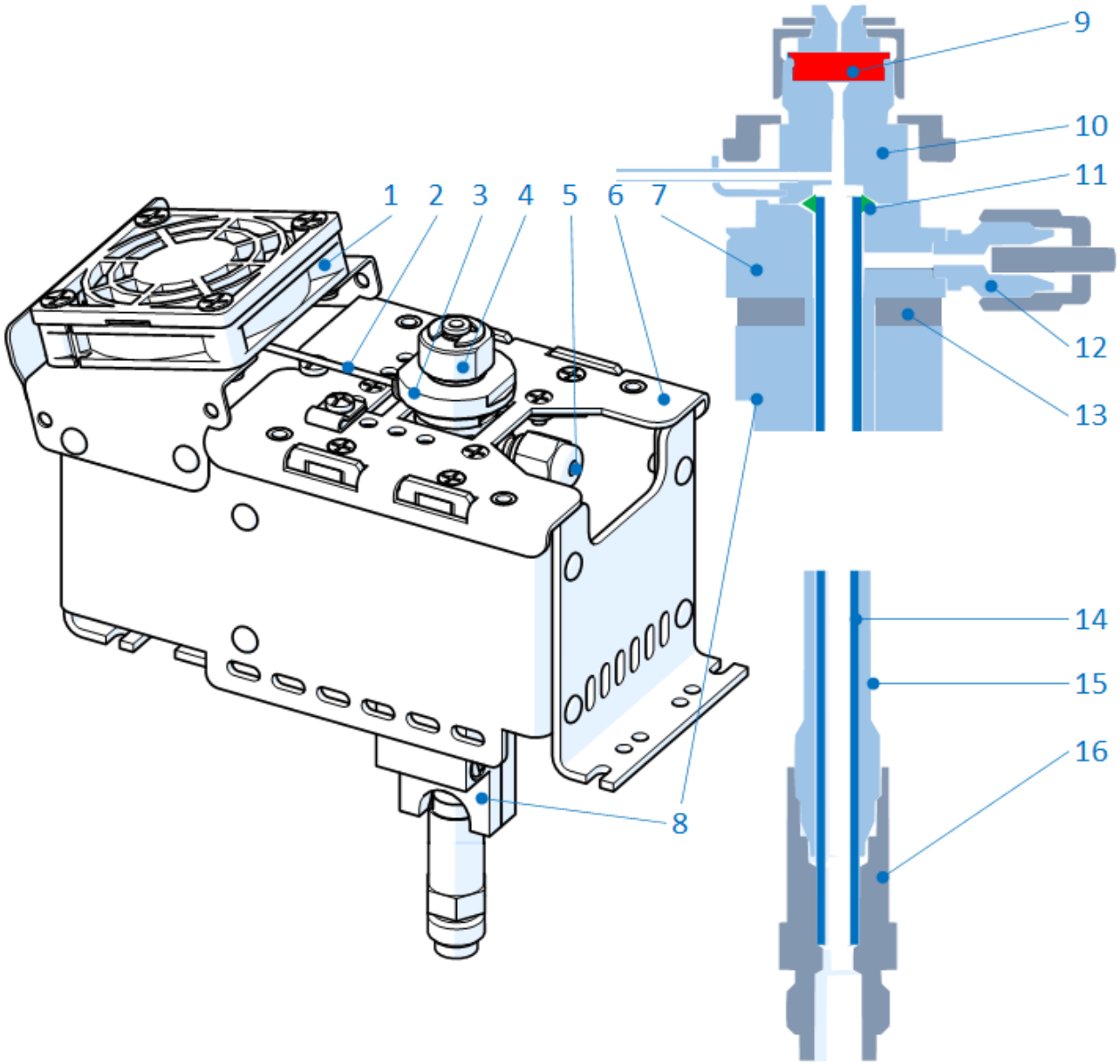
Liner 14, located in the body, is a glass tube of 120mm length. Inside the liner, vaporization of the sample occurs. Liner is sealed with rubber O-ring 11.

An adapter is installed in the upper part of the inlet body, fixed with a nut 3.

Sample injection is performed through the septum 9 retained by nut 4 on the adapter.

A fan mounted on the power casing cools the adapter and the septum.

Thermal washer 13 may be set in (from shipping kit), if high temperature of the top part of the liner is required in order to order to avoid losing high boiling components.



1 – Fan, 2 – carrier gas tubing, 3 – adapter nut (8.930.222), , 4 – septum nut (6.482.013), 5 – cap (6.433.021), 6 – cover (8.430.487), 7 – inlet body (6.113.097), 8 – heater (8.020.004), 9 – septum,10 – adapter (5.889.022-01), 11 – rubber liner sealing (O-ring) (8.683.032),, gas tubing, 12 – vent fitting (sealed), 13 – thermal washer (8.940.007), 14 – liner, , 15 – bottom fitting of the inlet body, 16 – packed column fitting (8.652.376) depends on column type.

Figure 1.2 – Inlet 7.2 (packed) design (P/N 150-1100)

1.3.2 Liner installation

It is recommended to replace the liner before starting work with the GC turned off and the inlet and the column oven cooled.

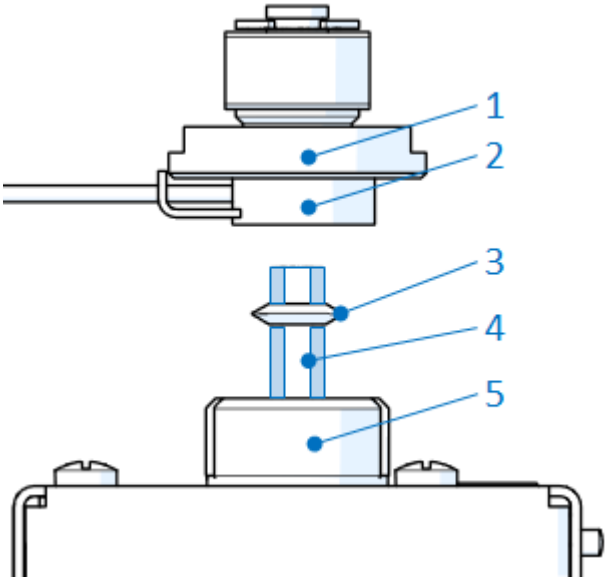


Touching inlet top parts, working at temperatures above 300 °C, may cause burns.

Liner installation is shown below.

- Before liner installation, put rubber O-ring 3 onto liner 4 at a distance (4-5) mm from the top edge.
- Then liner is positioned into body 5 of inlet and pressed from above by adapter 10. The adapter is kept by nut 1 screwed onto inlet body.

When tightening the inlet adapter nut and the septa nut, do not apply excessive force. Usually, the effort of tightening by hand is enough.



1 – nut, 2 – adapter, 3 – O-ring, 4 – liner, 5 – inlet body

Figure 1.3 – Liner installation

1.3.3 Pneumatic connections

DFC10 connection of packed inlet:

- Precise mass flow is provided even at oven temperature programming.
- Other carrier gas value set is possible for blowing stage.
- Set value: DFC10 – flow rate.

DPC11 connection of packed inlet:

- Minimized pressure surge at sample vaporization.
- Sample injection at pulsed pressure mode is available.
- Programming carrier gas pressure.
- Set value: DPC11 – pressure.

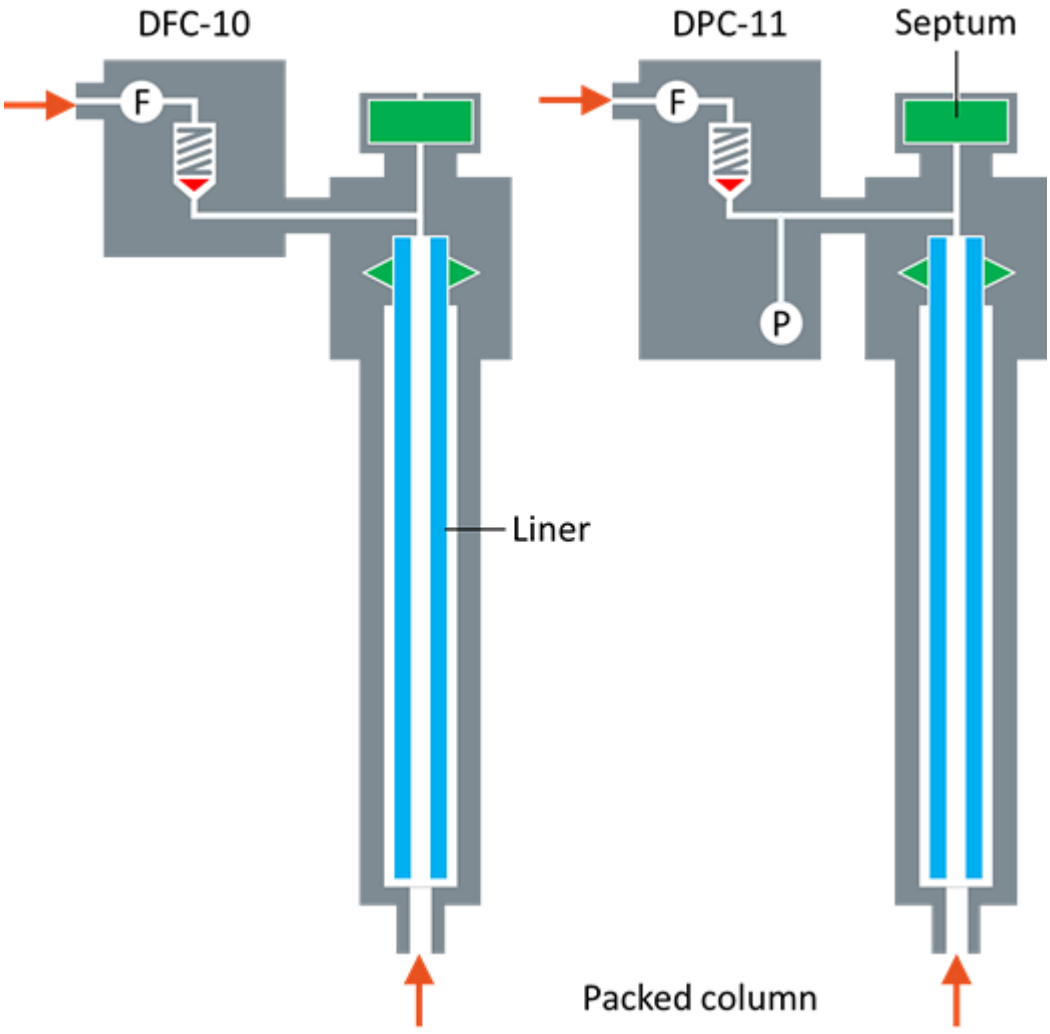


Figure 1.4 – Packed inlet (P/N 150-1101) connection of DPC10

1.3.4 Packed column connection

Preparation and installation of typical packed columns (type 1, symmetrical) is described in details in section 4.1. Packed column preparation procedures, Part 1 UM. In this case standard fitting 8.652.376-02 is applied (Figure 1.2).

Installation of column with elongated front end (type 2)

Column with elongated front end (Figure 1.1) allows to provide injection directly into packed column in order to reduce solvent peak tailing. Liner should be preliminary taken out from the inlet.

- Put nuts and ferrules onto the column.
- Unscrew fitting 16 (Figure 1.2) and attach fitting 8.652.376-02-06 instead (supplied by order). Fitting is screwed with force “by hand”, then further tightening by wrench.
- Insert the long end of the column through the inlet, then put rubber O-ring 11 on distance (4-5) mm from the top end, in the same way as for liner.
- Screw the nut (at detector side) with force "by hand".
- Then fix the adapter 10 and nut 3 holding it on the top of the inlet.
- Screw the nut (at the inlet side in the oven) with force "by hand".
- If necessary, left and right nuts of column are screwed further with wrench (for graphite ferrules required).

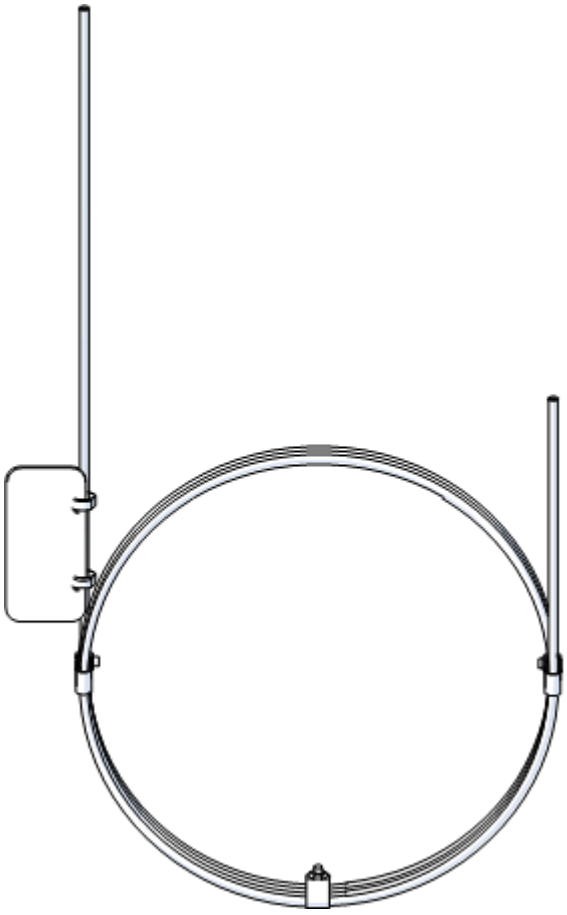


Figure 1.2 – column with elongated front end (type 2)

1.4 Capillary Inlet (split/splitless) 7.2 (P/N 150-1200)

Capillary Split/Splitless inlet (SSL inlet) is intended for injection of samples into capillary column of internal diameter from 0.05 to 0.53mm using split or splitless injection mode. The inlet has electronic pneumatic control, see details p.1.4.4. If necessary, the inlet can be reconfigured to work with packed column (see details p.1.4.4).



Touching inlet top parts, working at temperatures above 300 °C, may cause burns.

The inlet body, with a heater installed on it, is connected with the power casing 6 by the upper part. The power casing allows you to orient the inlet body relative to the vertical axis. The design of the power casing is the same for all versions of inlets.

Inlet body has vent fitting 12, to which split vent line may be connected if packed inlet is inverted into split/splitless inlet.

Fitting sealed with cap for packed inlet. In the bottom part, a part 18 (insert) is installed on the inlet body to connect the capillary column.

Liner 14, located in the body, is a glass tube of 120mm length. Inside the liner, vaporization of the sample occurs. Liner is sealed with rubber O-ring 11.

An adapter is installed in the upper part of the inlet body, fixed with a nut 3. The adapter 10 has two oppositely arranged tubings: carrier gas 2 and blowing septum tubing 5. The difference between them is blowing septum tubing 5 is shorter than the carrier gas tubing 2.

Sample injection is performed through the septum 9 retained by nut 4 on the adapter.

A fan mounted on the power casing cools the adapter and the septum.

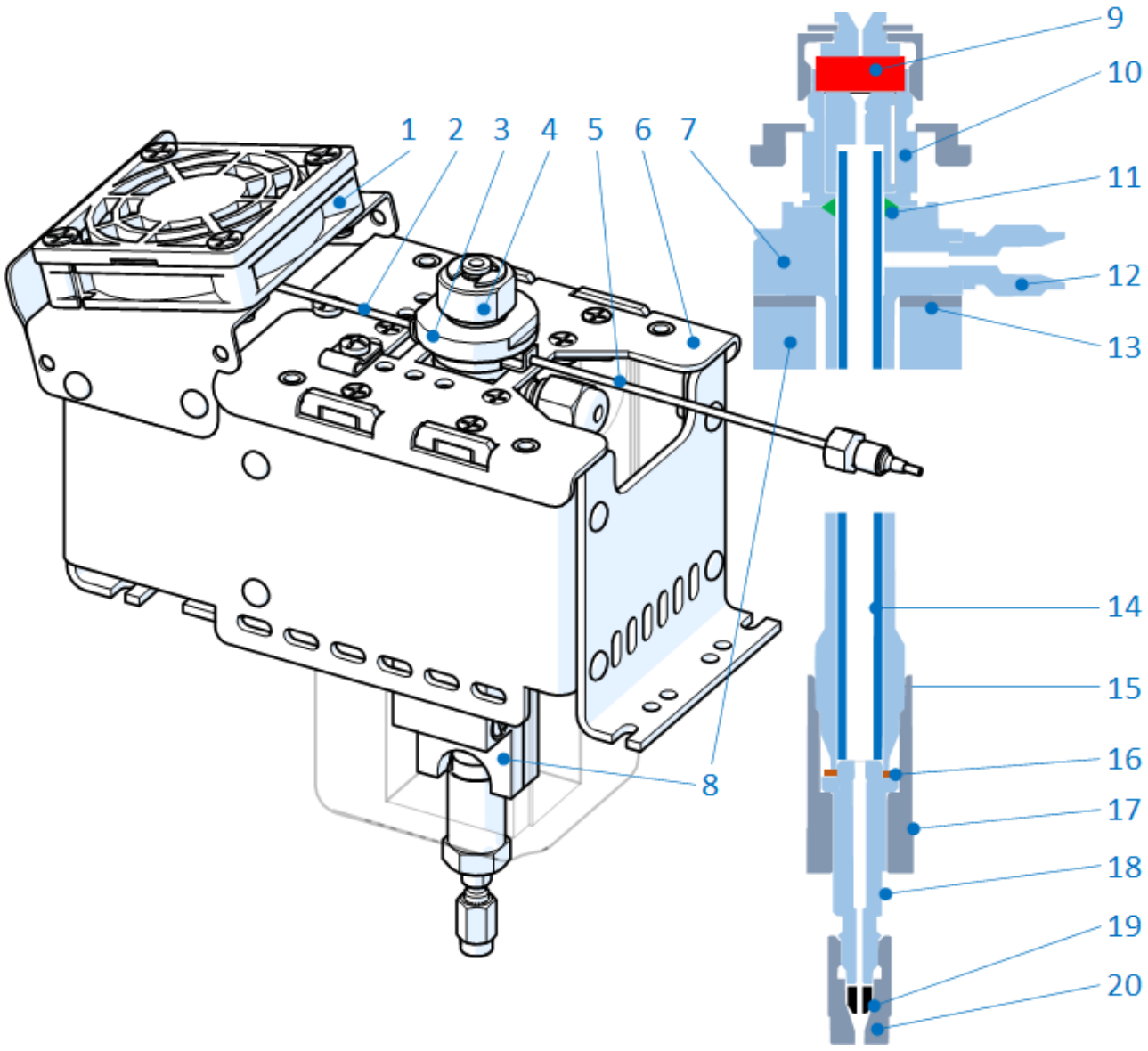
DPC 11.A2-2 (B2-02) is connected to tubing 2 to supply carrier gas to the inlet. Septum blowing flow provider is connected to tubing 5, which can be a mechanical pressure controller 5.157.012 (see Figure below) or an electronic controller DFC10D-02 (supplied by order).

Thermal washer 13 may be set in (from shipping kit), if high temperature of the top part of the liner is required in order to order to avoid losing high boiling components.

The quick-detachable version of the 7.2 capillary inlet contains electrical connectors (heater and temperature sensor) located in the accessible part of the analytical platform.

1.4.1 Capillary Inlet design

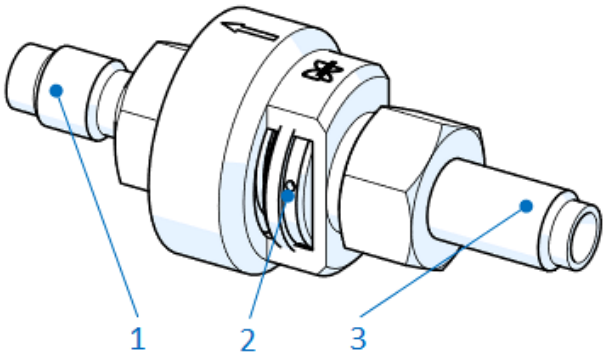
Do not touch the inlet top part (nuts 3, 4 9) at heated inlet. Touching can cause burns. Maintenance service of inlet should be carried out after its cooling to temperature below 50 °C.



1 – Fan, 2 – carrier gas tubing, 3 – adapter nut (8.930.222), 4 – septum nut (6.482.013), 5 – blowing septum tubing, 6 – cover (8.430.487), 7 – inlet body (6.113.097), 8 –heater (8.020.004), 9 – septum, 10 – adapter (5.889.021-01), 11 – rubber liner sealing (O-ring) 8.683.032, 12 – vent fitting, , 13 – thermal washer (8.940.007), 14 – liner, 15 – bottom fitting of the inlet body, 16 – washer (8.940.008), 17 – adapter nut (8.652.685) for capillary column, 18 – Insert (6.278.006 for liner 99mm length or 6.278.006-01 for liner 113 mm length), 19 – ferrule, 20 – capillary column nut (8.930.339).

Figure 1.5 – Capillary inlet 7.2 (split/splitless) (P/N 150-1200) design

Mechanical pressure controller 5.157.012 has a built-in pneumatic resistance and can maintain a flow rate of (1-5) ml/min at an inlet pressure of the regulator from 10 to 1000 kPa. The direction of gas movement through the regulator is indicated by an arrow on the housing. Adjustment of the gas flow through the MPC is carried out by rotating the adjusting element. The regulator is installed in the blowing channel of the inlet septum.



1 – Outlet, 2 – adjusting element, 3 – inlet

Figure 1.6 – Mechanical pressure regulator 5.157.012

Septum blowing is required to prevent ingress septum destruction products into carrier gas flow and GC column. Optimal flow rate for septum blowing is (2-5) ml/min. Digital flow controller DFC10D-02 allows programmable setting of septum purge flow for optimized operation in different injection modes and gas saver mode.

For GC transporting purpose, blowing septum tubing (4) is fixed with clamp on the cover (6). After GC has been installed at workplace, it is recommended to release screw (Figure 1.8) clamping tubing (4). Otherwise, removing adapter (9) from inlet is impossible. For transport purpose tubing is to be fastened again.

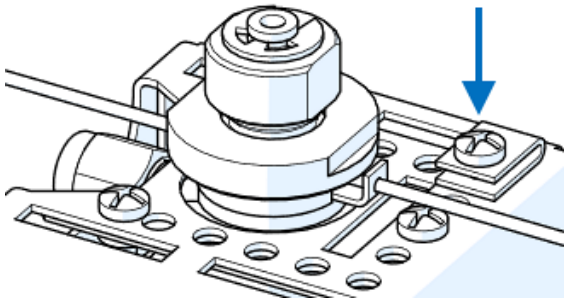


Figure 1.8 – Release of septum blowing tube from transport fixation

1.4.2 Liners for SSL inlet

Liner 14 (Figure 1.5) is a chamber for vaporization liquid sample.

The following liners are available for inlet 7.2:

- Liners of 113 mm length (Table 1.), shortened insert 17 (6.278.006-01) is to be used in this case,
- Liners of 99 mm length (Table 1.) (suitable commercially available liner for SPL–14 inlet Shimadzu GC 14/15A/16), extended insert 6.278.006 is to be applied.
- Liners of 95 mm length, not described in this document, they are available by catalogues Restek, SGE. The Insert 6.278.006-02 should be applied for 95 mm liners (available by order).

Liner is a quartz or borosilicate tube, having 5mm outer diameter, and 113 or 99mm length. For the better vaporization process, a wad from glass fiber, glass wool or other inert material may be inserted into liner (filling). Overview of available liners for inlet 7.2 is given below. O-ring 8.683.032 is not included in the liner at delivery.

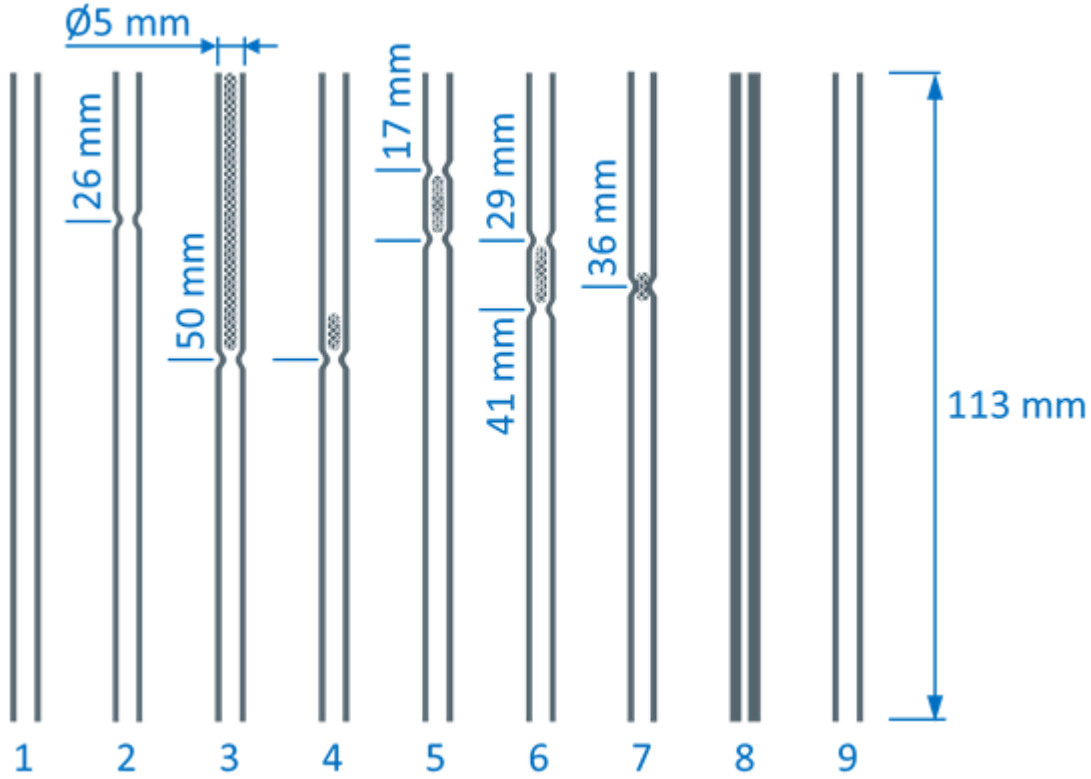


Figure 1.8 – Liners 113mm length

Table 1.7 – Liners 113mm length

#	P/n	Filling	Application
1	7.352.054	no filling	General applications. For use with frequent replacement of glass wool or fiber. For manual or automatic injection.
2	7.352.057	no filling	It is recommended for analysis of alcohols by GOST R 51698 and GOST R 51786. For automatic injection[1]
3	6.236.034	glass fibers tampon	Injection into capillary column. For general applications. For manual or automatic injection.
4	6.236.034-01	quartz fiber	Injection into capillary column. For analysis of samples containing temperature-unstable components. For manual or automatic injection.
5	6.236.078	glass fibers tampon.	Injection into capillary column. Rated restriction. It is recommended for analysis of alcohols and other liquid samples forming great volume of gas at evaporation. For automatic injection[1]. There are restrictions on the use[2].
6	6.236.078-01	glass fibers tampon.	Injection into capillary column. Rated restriction. It is recommended for analysis of alcohols and other liquid samples forming great volume of gas at evaporation. For manual injection. There are restrictions on the use[2]
7	6.236.078-02	quartz fiber	It is recommended for analysis of alcohols by GOST R 51698 and GOST R 51786. For automatic injection
8	7.352.066	no filling	To reduce bandwidth zone widening when operation with external samplers: Thermal desorber, headspace sampler, liquefied gas sampling valve.
9	8.236.430	no filling	Metal. For on-column injection into the capillary column. Assembly (spring, insert, liner) 6.233-01

[1] autosampler AS–2M with needle 43 mm.

[2] Application notes for the analysis according to GOST 30536-2013 (GOST R 51698) (link to the website of SDO "Chromatec"). The liners recommended for analysis are additionally listed in the operating manual for analysis.

Table 1.8 – Liner 99mm length

Application	Cat.N (Restek)
The liner for Splitless-mode. Analyses of trace concentrations. Can be filled with glass wool. For general applications, can be applied to split-mode. Volume 640 mm3.	#20860 or #09200831 SGE

113 mm liners application features

Liner 7.352.054 (without filling) is applied in the following cases:

- Gaseous or easy evaporable liquid sample is to be injected into inlet, taken that it comes into GC column with reproduced constancy;
- Sample does not contain nonvolatile or semi volatile components, capable to pollute chromatographic column;
- At connecting external peripherals to inlet, such as thermal desorber, headspace sampler, liquefied gas sampling valve, pyrolizer. In this case liner 7.352.066 with lower internal volume can be recommended as well.
- Filling material (glass wool) reacts with target compounds of the sample, so liner with filling is not suitable.

This liner is quite convenient in operation when sample is very dirty and glass wool is to be changed frequently.

Liner 6.236.034 has a "neck" for stable location of the filling in chamber. The filling (glass wool or fibers) is placed above "neck" in the level of the needle end location at injection by syringe (51 mm length). Filling in the liner provides efficient vaporization and mixing of the sample in gas flow when operating in split mode.

The liner is typically used in following cases:

- Injected liquid sample may contain nonvolatile or semi volatile components, capable to pollute the chromatographic column;
- Due to sample character, it’s impossible to obtain good repeatability at sample injection into empty liner;
- Inlet works in split / splitless mode;
- Filling material is inert to sample compounds. If necessary, change filling material (see below).

Liner 6.236.078-01 has a narrow "neck" for stable positioning of syringe needle in liner filling at injection (51mm needle length). Recommendations on application of this liner are similar to the previous one. Distinctive point: liner is recommended for injection liquid samples forming great gas volume at evaporation.

Liner 6.236.078 the same as liner 6.236.078-01, intended for operation with autosampler AS-2M with syringe needle length 43mm. The filling is positioned higher comparatively to liner 6.236.078-01.

Selection of filling material. Keep in mind that the filling may cause peaks tailing in some cases, especially when it’s too tight. In other cases, the filling may be a source of sorption or destruction of analytes. So, filling selection and usage must be strictly determined by application.

Filling material must be chemically inert and have no tend to sorption of analytes.

Standard liner at delivery is filled by glass fibers tampon, similar to untreated glass wool in property; it's recommended for analysis of polar and non-polar volatile compounds.

For analysis of traces of pesticides, drugs and other semi-volatile organic compounds, silanized glass wool or quarts wool is recommended.

1.4.3 Liner installation (replacement)

It is recommended to replace the liner before starting work with the GC turned off and the inlet and the column oven cooled.

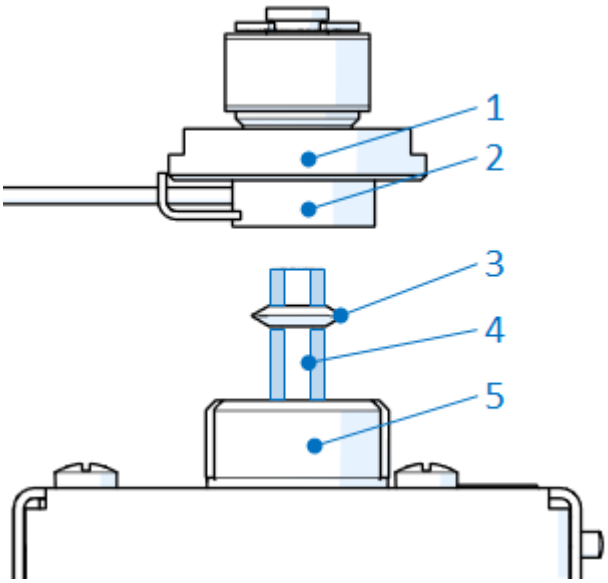


Touching inlet top parts, working at temperatures above 300 °C, may cause burns.

If liner of another length is to be used, change the insert 17 (Figure 1.5).

Liner is to be installed (replaced) as follows:

- Before liner installation, O-ring (8.683.032) 3 should be put on liner 4 and moved on distance (1-2) mm from the top edge.
- Disconnect adapter from the inlet. For this unscrew nut 1 and remove adapter carefully.
- Using hook from shipping kit or pincers take out old liner.
- Then insert new liner into inlet body 5. Liner should loosely rest on insert 17 (Figure 1.5).
- Put adapter 2 with nut 1 on the inlet. Nut being screwed on the inlet body, will push rubber O-ring down, so liner keeps pressed to the insert.



1 – Nut, 2 – adapter, 3 – O-ring, 4 – liner, 5 – inlet body

Figure 1.7 – Liner installation

When tightening the inlet adapter nut and the septa nut, do not apply excessive force. Usually, the effort of tightening by hand is enough.

- After performing the procedure, turn on the chromatograph to transfer the instrumental method, it is recommended to set the temperature of the column oven at room level for 10 to 15 minutes to blow off the oxygen trapped in the inlet after septum replacement.
- Check the tightness of the inlet in accordance with section 1.7.1.
- If a liner filled with fiber glass is used, it is recommended to bake it at high temperature (up to 270 – 300 ° C) and a large sample split (> 1:50) for 30-60 minutes to remove volatile contaminants. After baking the liner, it is necessary to perform blank analysis without entering a sample, to make sure there are no false peaks.

1.4.4 Pneumatic connections

Digital gas control is provided for inlet. For SSL inlet P/N150-1201 Digital pressure controller DPC11A2-02 (or high-pressure controller) supports set pressure level at input of the inlet. Digital flow controller DFC10-02 (or high-flow controller) supports flow level at split vent (outlet), regulates split ratio. Either mechanical gas controller (on default) or electronic DFC10D-02 (by order) controls septum purge flow. DPC11 controller maintains the set pressure at the column inlet. Vent gas controller and septum blowing work according to a given program, maintaining the flow rate in accordance with a given methodology. The connection diagram of the gas controllers to the capillary inlet is shown below.

DPC11 provides fine pressure control at the nearest point to column inlet thanks to pressure sensor with separate fitting; this allows obtaining excellent retention times repeatability.

Adsorptive trap 5.884.048 (vent trap) is installed in vent line to clean vent gas flow from solvent vapor. The valve 5.890.042-04 (vent valve) prevents contaminations coming from vent trap into inlet, installed optionally.

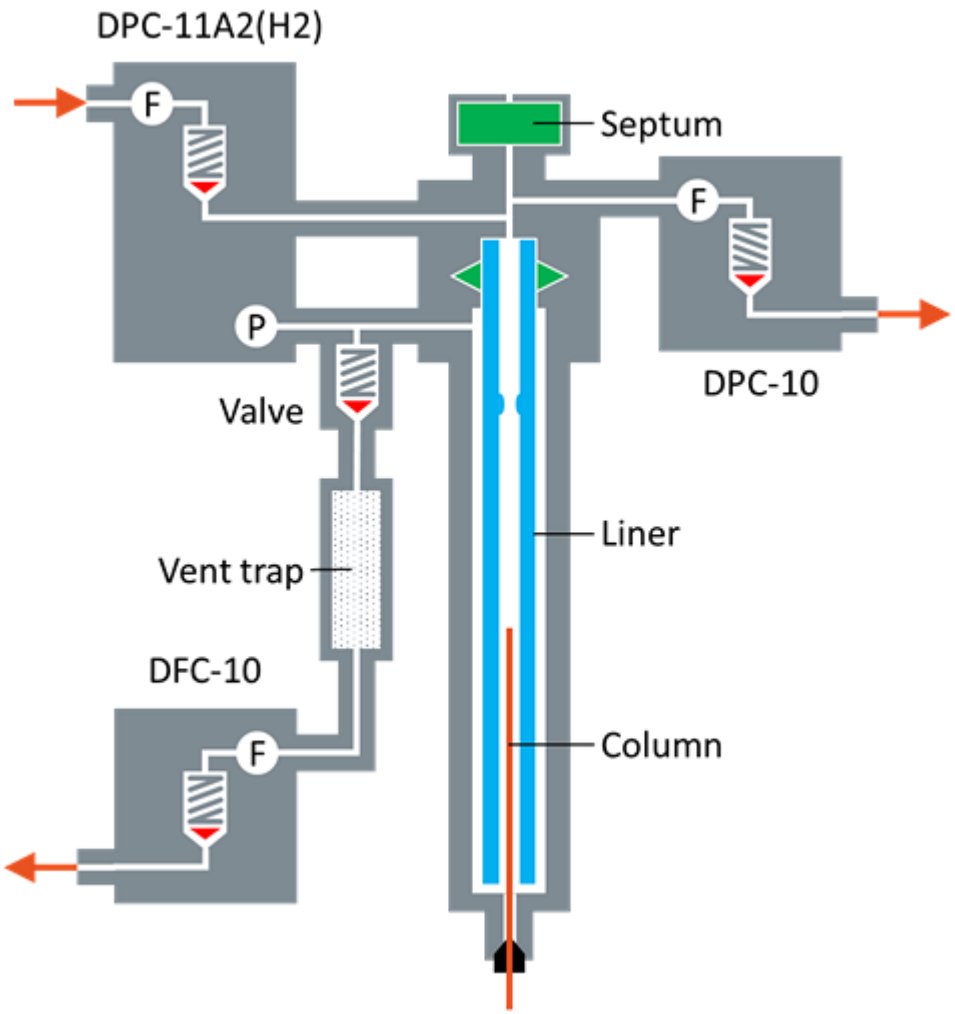


Figure 1.11 – Split/splitless inlet (P/N150-1201) pneumatic connections

Another pneumatic diagram (Figure 1.8) adopted for operation with wide bore columns is referred to Split inlet P/N 150-2102. Digital flow controller DFC10H-02 supports total flow level at input of the inlet. Digital pressure controller DPC11U-02 provides upstream pressure control at the column inlet and split ratio. Either mechanical gas controller (on default) or electronic DFC10D-02 (by order) controls septum purge flow.

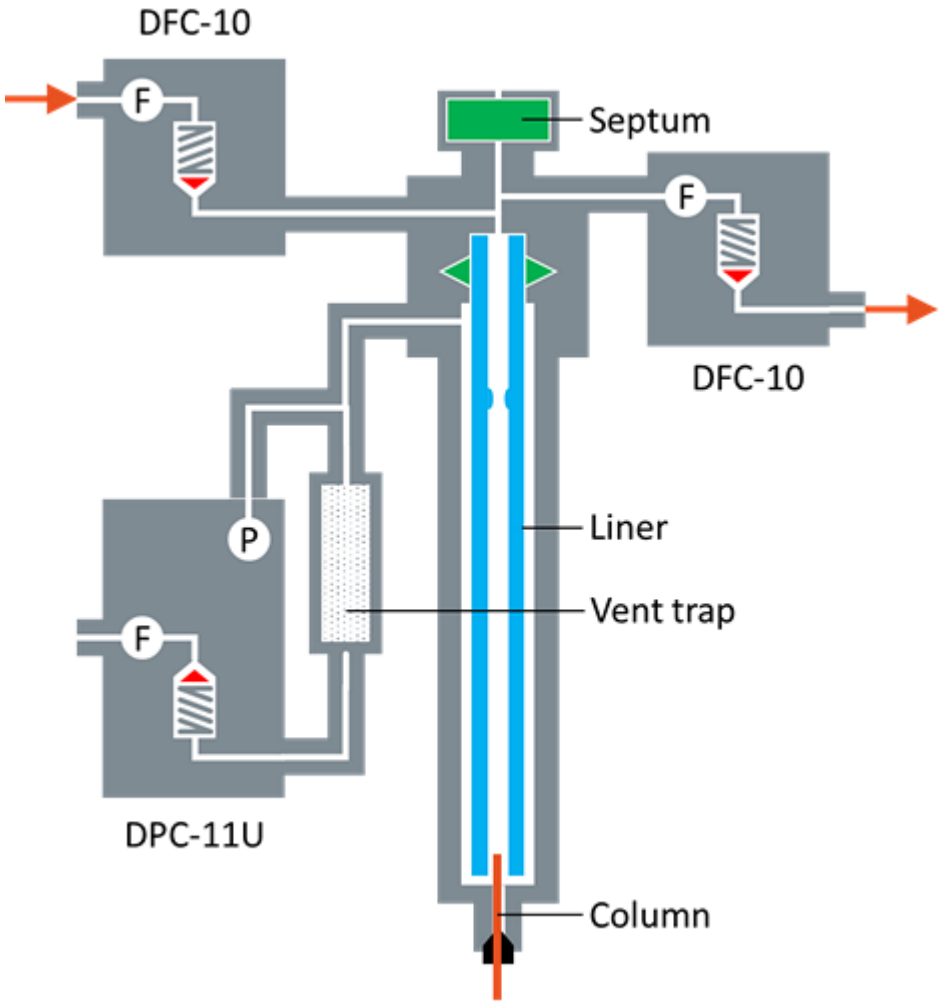
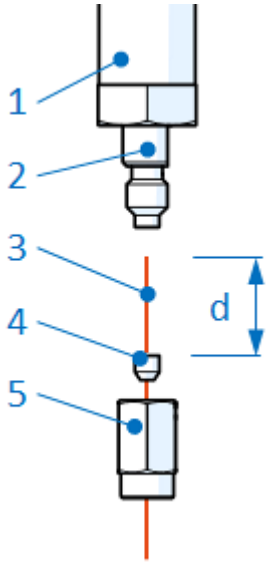


Figure 1.8 – Flow diagram. Split inlet (P/N150-1202) for wide-bore columns

Information on the types of flow and pressure controllers is given in UM Gas chromatographs. Part 1. General information.

1.4.5 Capillary column connection

Capillary column is sealed in the fitting of the inlet with the help of graphite ferrule 18 and nut 19.



1 – Nut (8.652.685), 2 – Insert (6.278.006 for liner 99mm length or 6.278.006-01 for liner 113 mm length, or 6.278.006-02 for liner 95 mm length), 3 – column, 5 – capillary column nut (8.930.339)

Recommended distance from ferrule to column end:

d=32±1mm for liner 113mm

d=43±1mm for liner 99mm

Figure 1.9 – Capillary column installation

The procedure of column installation to inlet:

- Put on the nut 5 and the ferrule 4 (e.g. #072626 SGE, with its cone into nut) on the end of column to be installed into inlet.
- Cut column end at (5–20 mm). This prevents column from graphite ingress. The cut should be flat without chips and scratches.
- Wipe cut end of the column with lint-free tissue wetted in alcohol in order to remove dust and other contaminants;
- Locate nut 5 and ferrule 4 at required distance (d) from the column end. For convenience, use column measuring tool 6.236.249 from shipping kit. Install column end into measuring tool, align column end at mark (1) for 113mm liner or (2) for 99 mm liner, pre-swage the nut in the tool fitting so that column is fixed tightly by ferrule. Then release nut.
- Insert the column into inlet adapter, tighten nut by hand.
- Then tighten the nut by wrench at 45-60 degrees so that column cannot be withdrawn from the fitting (column is tightened by ferrule holder).

Once the column is connected to inlet it should be blown by carrier gas from oxygen at ambient temperature at least for 10-15 min.

1.4.6 Inlet gas flow control

DPC-11H2-02 allows pressure programming settings:

- constant pressure or programming ramps at column inlet;
- pressure, speed or cap.flow units through capillary column;
- pulsed injection.

Split vent flow gas controller (DFC-10) supports setting:

- split ratio or split vent flow;
- split or splitless modes;
- carrier gas save mode.

DPC-11H2-02 forms pressure at column inlet. When operating in the speed and flow modes, these values are automatically converted into pressure. The recommended flow of carrier gas through the column, depending on the type of gas and the inner diameter of the column is given below ((Table 1.).

Gas controller DFC-10D-02 at septum purge channel (if available) sets gas flowrate and turns it off in gas save mode and in splitless injection mode.

Table 1.9 – Optimal flow rate for capillary columns

Column internal diameter, mm	**Carrier gas flow, ml/min**		
	Hydrogen	**Helium**	**Nitrogen**
0.05	0.3 – 0.5	0.2 – 0.3	–
0.1	0.5 – 0.8	0.3 – 0.5	–
0.18	0.7 – 0.9	0.5 – 0.7	0.35 – 0.5
0.25	1.3 – 1.8	0.8 – 1.3	0.6 – 1.0
0.32	2 – 3	1.5 – 2	1 – 1.5
0.53	6 – 8	4 – 6	3 – 4

1.4.7 Split injection operating parameters

Split injection is mainly applied for analyses of components in high concentration (≥50 ppmw).

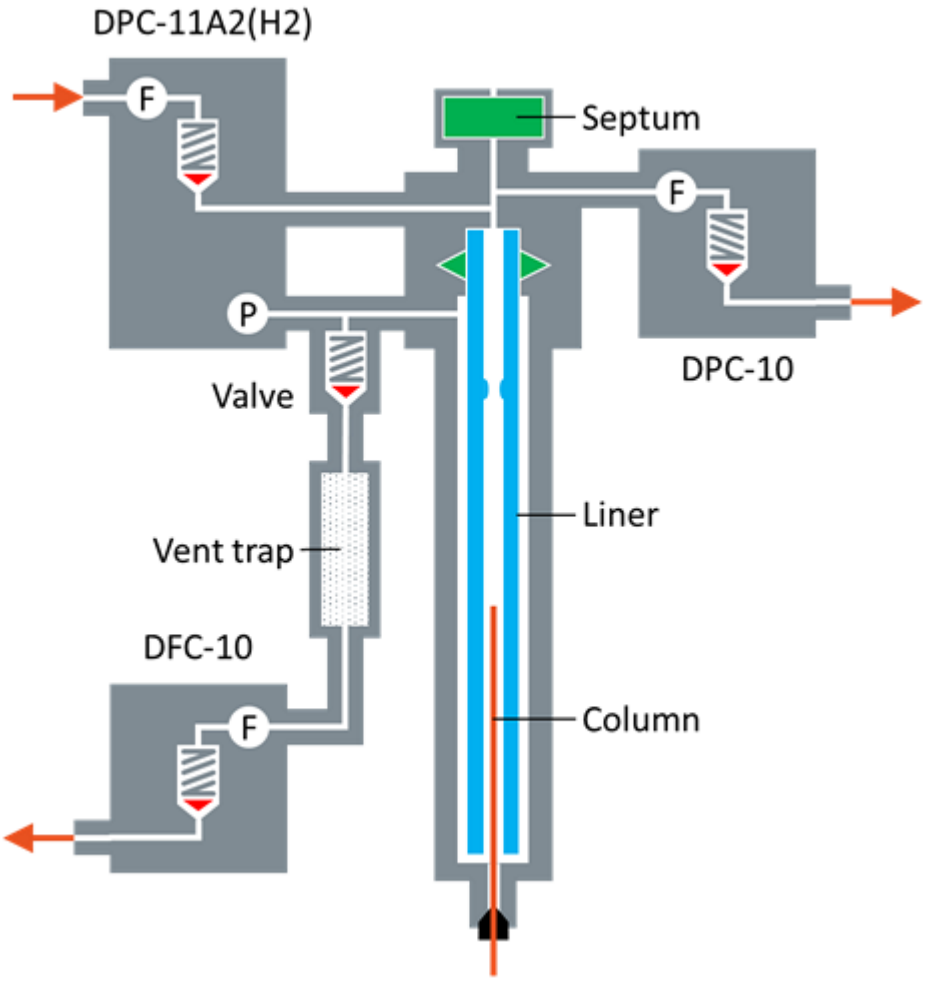


Figure 1.10 – Inlet flow diagram in split mode

When operating in split mode, sample is injected in heated inlet and instantly evaporates. Small part of the sample comes into column, the rest of sample is flown away through vent gas line according to appointed split ratio. For the best injection repeatability at split mode, the sample should be evaporated quickly and effectively mixed with carrier gas. Liners of various designs are applied for this purpose (see section 1.4.2).

Recommendations on GC mode for split injection are suggested below.

Inlet temperature for split mode is usually set on (50-100)°C above than boiling temperature of highest boiling analyzed component. It is necessary to avoid excessively high temperatures of the inlet. It can result in arising ghost peaks – low-molecular products of inlet septum destruction, or chemical transformations of sample components.

Volume of injected sample in evaporated state should not exceed volume of the liner (0.8 ml for 113mm liner). It is necessary to get as narrow sample's zone as possible at the beginning of the column. The maximum volume of injected sample depends on solvent properties (molecular weight, boiling temperature), working temperature and pressure in the inlet. For water the maximal sample volume does not exceed 0.7 ul, for methanol, ethanol, acetonitrile – 1.5 ul, for acetone, diethyl ether, chloroform – 2 ul, for hexane, toluene, isooctane – 4 ul.

Optimum **working parameters of input pressure controller** (pressure, speed or cap. flow) depend on carrier gas type, column parameters and method requirements.

Split ratio for columns of (0.25–0.32) mm i.d. usually is 1/15 … 1/100, for wide bore columns (0.53 mm i.d.) is from 1/5 to 1/20, for narrow columns (0.15–0.2) mm i.d. is from 1/40 to 1/200. To avoid peaks broadening in the inlet, low split ratio is recommended to combine with analytes focusing in the beginning of the column.

Split vent flow controller parameters setting can be carried out either by setting flow rate directly or setting split ratio which will recalculated into flow rate then.

Column temperature. Both isothermal mode and temperature programming of the oven can be applied.

Gas saver. It is recommended to combine split sample injection at large values of sample vent flow with carrier gas saver. To use the gas saving function when setting the instrumental method, select sample injection mode: "Split + gas saver". Carrier gas flow at the saving state (the last value of flow) is recommended to be set at least 10 ml/min in order to avoid the injection of volatile substances from vent line into the column between analyses and, as a result, the appearance of false peaks. When using gas saver and manually injecting sample, the chromatograph will switch to the "Waiting" state after all parameters return to normal, to switch to the "Ready" stage, press the START button. For more information about the "Waiting" state, see Section 3.7.2, Part 1 of the UM.

1.4.8 Splitless Injection operating parameters

Splitless Injection is applied for analyses of components in low concentration, at splitless injection the whole sample is evaporated and transferred into capillary column. The splitless mode is acceptable, if analytes are eluted later, than solvent.

In order to prevent tailing of chromatographic peaks due to long sample pass from the inlet into column, the splitless mode is always combined with focusing of analytes in capillary column heading. Therefore in splitless mode only those components may be analyzed which are focused at beginning of the column.

Splitless Injection includes the following stages:

- Before sample injection the valve of controller DFC-10 at vent channel, vent valve and DFC-10 at septum purge (if available) turn to close position. See flow diagram

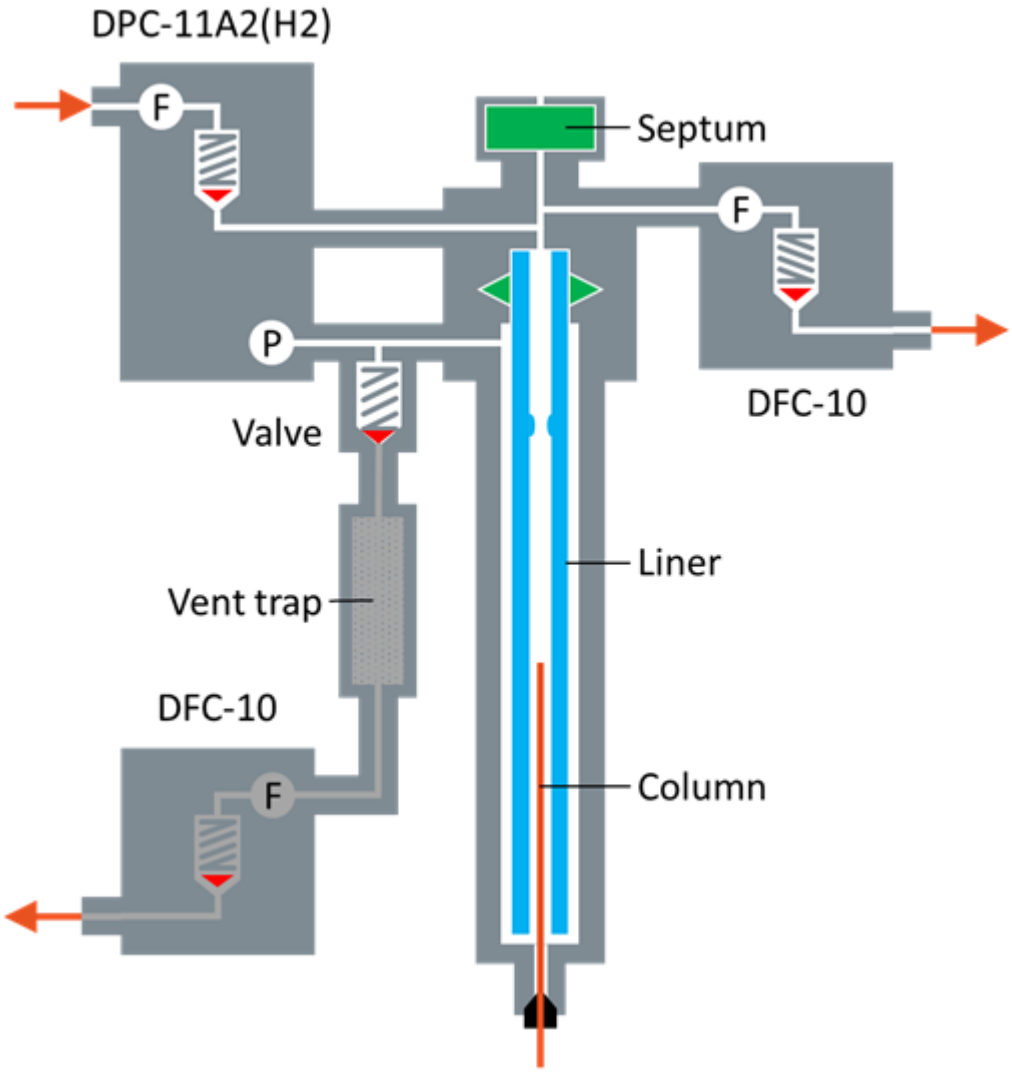


Figure 1.11 – The temperature of column is set so that analytes were focused in the column heading

- The sample is injected into hot inlet and gets evaporated.

- Within some time (0.5-1.5 min) sample vapors transfer from inlet chamber into column.
- At input in column the sample solvent is gradually carried away by gas flow, and analytes remain in column heading.
- Then valve of controller DFC-10 at vent channel, vent valve and DFC-10 at septum purge (if available) turn open, the remaining sample vapors are swept out from the inlet. Time of inlet blowing is (1 – 3) minutes. Upon termination of blowing the small vent flow rate (10-20) ml/min is set. Inlet flow diagram with opened vent valve is the same as for split mode.
- Column oven starts quick heating, analytes are eluted as narrow peaks.

To minimize spreading away from liner into vent channel vent valve 5.890.042-04 is recommended to install before adsorptive vent trap 5.884.048. The valve switches closed before sample injection and opens once sample transferred into GC column, at the same time as DFC 10 in vent line. If vent valve is not available in your GC, it's recommended to remove vent trap 5.884.048 when operating in splitless mode.

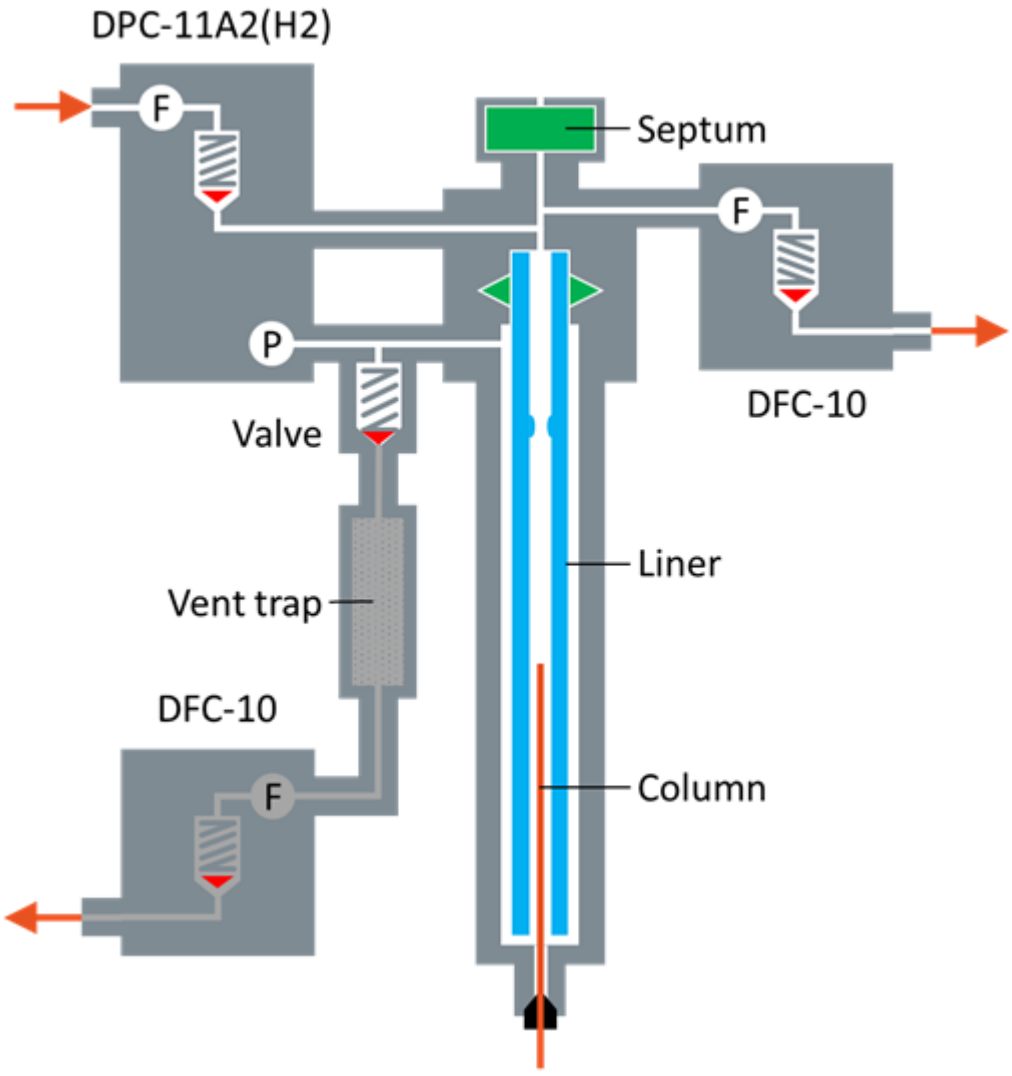


Figure 1.11 – Inlet flow diagram in splitless mode

Recommendations on GC mode for splitless injection are suggested below.

Liner. For splitless mode liners of 99 mm length are recommended, e.g. #20860, 20863 (by Restek catalogue) or #0920831 (by SGE catalogue). The filling is not recommended to place into liner, as it may cause analytes decomposition. To minimize sample dilution, narrower liners can be used, but take into account maximum evaporated sample volume, inlet chamber should be not overloaded.

Inlet temperature. With components rather for long time being in the liner, the inlet temperature can be lower, than at split mode. In most cases the inlet temperature has a range from 200 to 280 °C. The analyses of "dirty" sample demand higher temperatures.

Sample volume. A larger sample volume can be used than when split flow injection, but care must be taken to ensure that the vaporized sample is also fitted in the liner. When manual sample injecting sample used, the method of hot needle insertion is preferred (preheating the empty syringe needle in the inlet). The sample feed rate should not exceed (1-2) µl per second.

Column temperature. To reduce peaks broadening out for analytes with boiling temperature lower 150°C it is recommended to use the solvent effect. Solvent effect can be obtained if initial column temperature is (20-30)°C as less as solvent boiling temperature. At heavier analytes determination the first step of column temperature can be higher than solvent boiling temperature, but much lower than boiling temperatures of analytes. Programming of column temperature begins after inlet blowing from sample rests and proceeds with high speed (15-30)°C minutes.

Oven temperature and gas flow diagram at splitless mode operation and pulsed injection is shown below.

In this example DFC-11 operates in "pulsed" mode: increased pressure value in the inlet at sample injection allows effective transfer of the sample from the inlet into column. Once sample transfer has been terminated, pressure goes down to normal value.

Injection procedure

- If the GC is on the state "Waiting" press START to transfer into "Ready" state.
- Once "Ready" state occurs, split vent gas controller shuts off.
- Inject sample into inlet and press START. At manual sample injection the method of injection by hot needle is preferable (pre-warming the syringe empty needle in the inlet). Speed of sample injection should not exceed (1-2) ul per second.
- The GC passes to "Analysis" state.
- Within 0.5 – 1.5 minutes sample vapors pass from the inlet into GC column, then vent gas controller turns open, sample rest is flown away from the inlet.

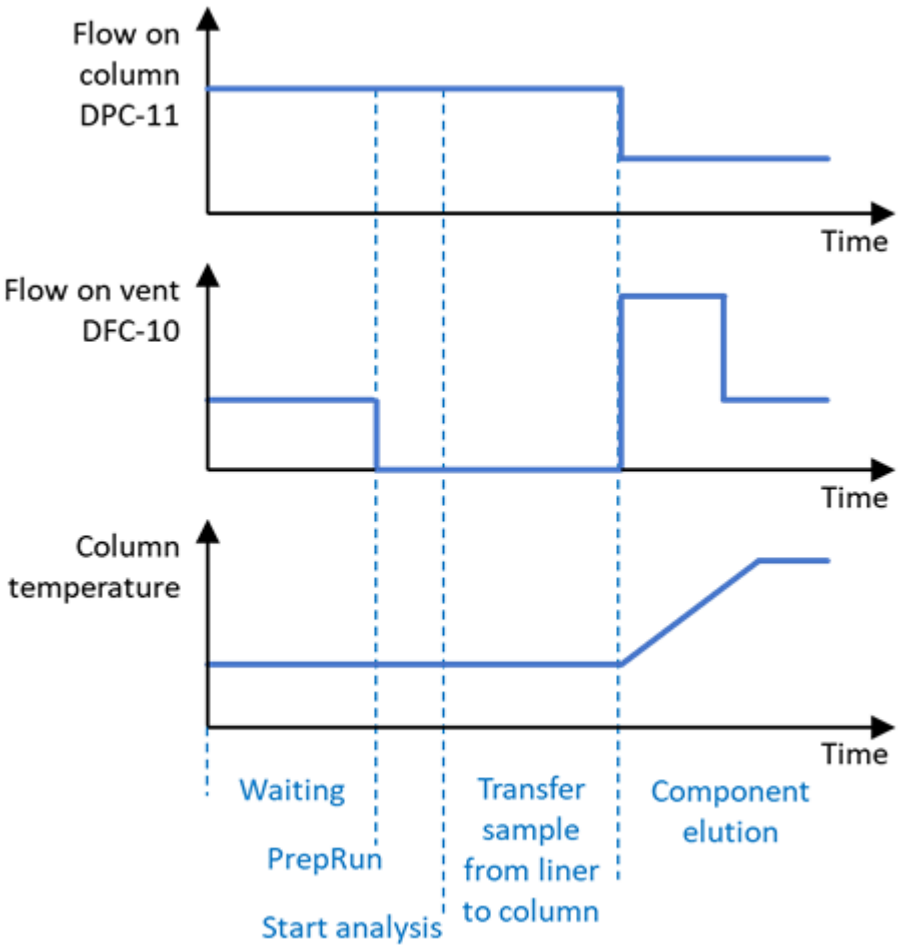


Figure 1.12 – Gases pressure/flow and column temperature at splitless mode

1.4.9 Split/splitless inlet usage with packed column

SSL inlet has design similar to packed inlet and may work with packed column. Before inlet disassemble oven should be cooled down, the GC is to be turned off, capillary column to be disconnected from the inlet.

Preparation of SSL inlet for packed column connection:

- In the oven: Disconnect adapter nut 17, insert 18, brass sealing 16 from the inlet (Figure 1.5). Instead attach adapter 8.652.376-02 from shipping kit of the inlet, intended for packed column connection.
- On the top: Unscrew nut 5 of adapter, remove adapter from the inlet, disconnect it from flow gas controller.
- Change liner if necessary.
- Fit another adapter 5.889.022-01 from shipping kit of the inlet. Recommended gas controllers' connection is shown in section 1.3.3.
- Vent fitting of the inlet should be sealed with plug 6.433.021 from shipping kit.
- Packed column connection is described in section 1.3.4.

1.5 Programmable Split/Splitless inlet 7.3, on-column inlet 7.4

1.5.1 Inlets design

Inlets 7.3 and 7.4 have a same design – body with a heater and a fan for fast cooling when working with temperature programming.

PS/SL inlet is intended for injection of samples into capillary column of internal diameter from 0.05 to 0.53mm. Inlet 7.3 provides 4 techniques of injection into capillary column:

- On-column injection
- Split mode;
- Splitless mode;
- Sample injection with pulsed pressure.

Inlet 7.4 is recommended for on-column injection.



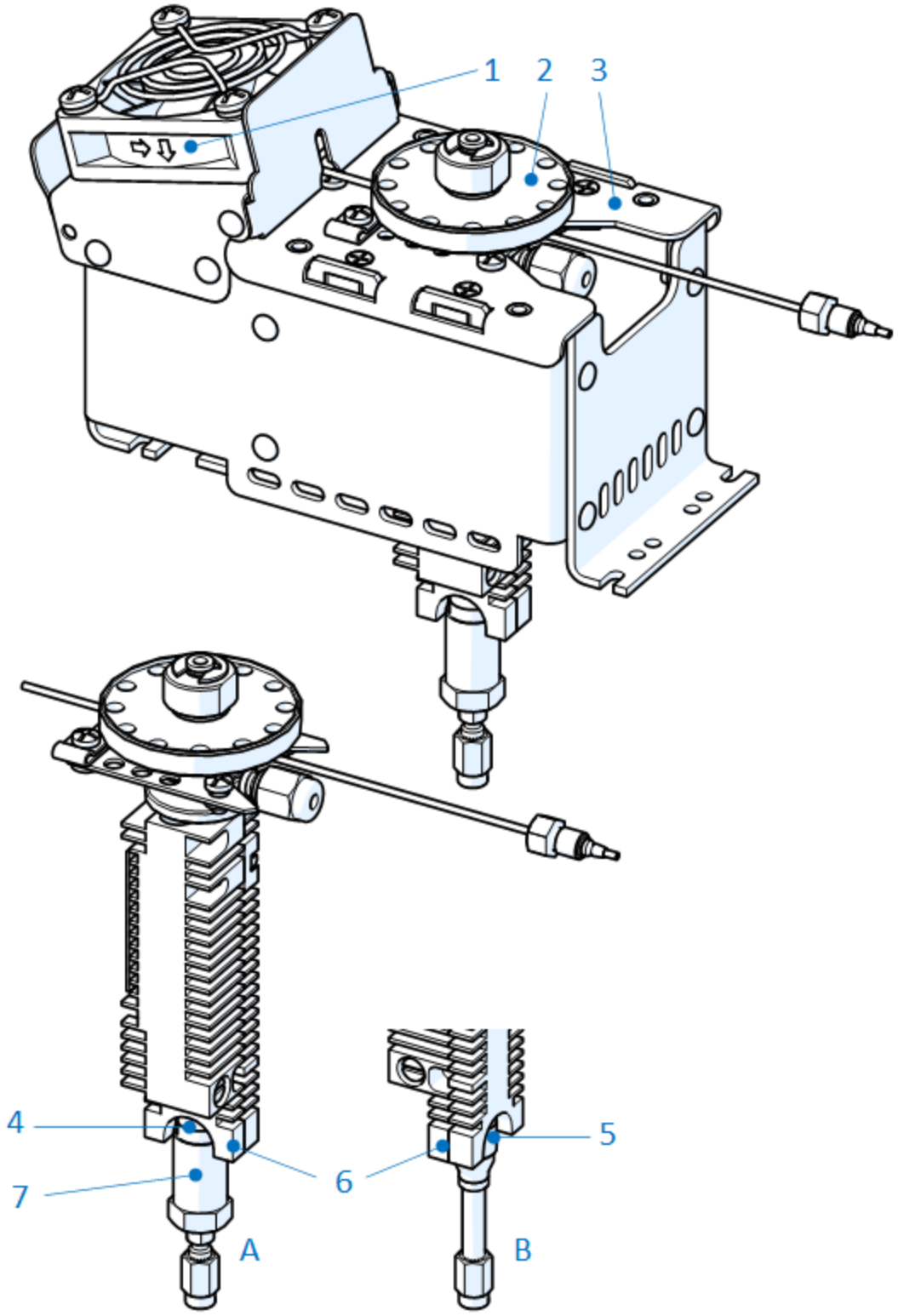
Touching inlet top parts, working at temperatures above 300 °C, may cause burns.

Maximum heating rate from 40 °C to 350 °C is 500 °C/min. Cooling time from 350 °C to 40 °C at 40°C in the oven is not more than 7 minutes. Cooling fan automatically turns on once “Analysis” state on the GC terminated.

The same liners are applicable for inlet 7.3 as for inlet 7.2. Gas connections to inlet 7.2 the same as for inlet 7.2.

With inlet 7.4 used only a metal liner 8.236.430 (in assembly (spring, insert, liner 6.233-01).

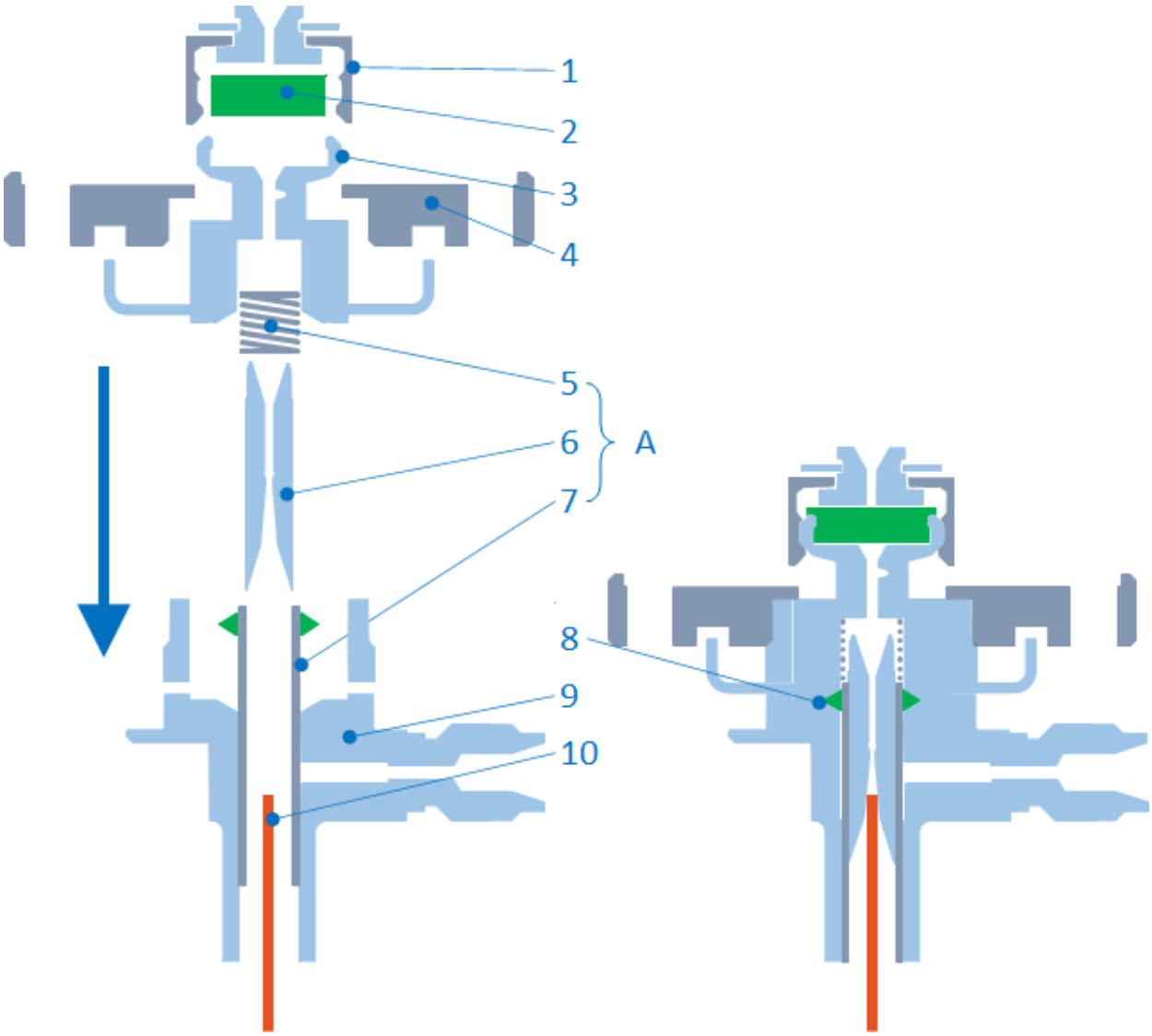
The fitting for connecting the column is non-removable for inlet 7.4.



A – inlet 7.3, B – inlet 7.4

1 – fan 5.883.013, 2 – nut 8.936.128, 3 – cover 6.430.487, 4– cower 6.113.097-01, 5 – cover 6.113.097-02, 6 – cover (8.035.341, 8.035.342), 7 – fitting 8.652.685 (removable)

Figure 1.3 – Inlets 7.3 and 7.4 design



1 – Nut 6.482.013, 2 – septum 7.010.019, 3 – adapter 5.889.075, 4 – nut 8.936.128, 5 – spring 8.383.068, 6 – sleeve 8.230.023, 7 – liner 8.236.430, 8 – O-ring 8.683.032, 9 – cover 6.113.097-02, 10 – capillary column

A – liner 6.236.233-01

Figure 1.4 – Inlets design 7.3 и 7.4 in section

It is recommended to use a special 11 mm VTO septa (or similar) from the spare parts kit of the inlet.

1.5.2 On-column operating parameters

On-column injection allows avoid component fractioning; therefore, this technique is recommended for analysis of samples containing components in wide boiling point range instead of split or splitless mode.

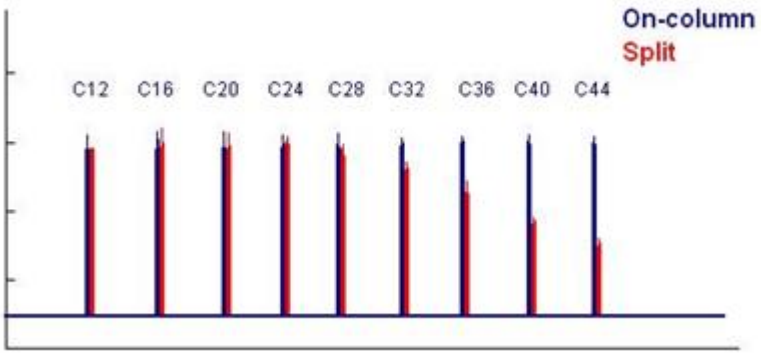


Figure 1.13 – Hydrocarbon peaks heights at on-column injection and at split mode

At on-column injection front end of the column is located in top part of the inlet so that needle of the syringe introduces directly into capillary.

Once sample is injected into capillary, inlet and column oven are heated by set programming, sample components elute and separate in the column.

Apparatus

Inlet for on-column injection should provide temperature programming at least in the rate of following oven temperature program.

Syringe for on-column injection should have elongated needle (70 – 100 mm), needle outer diameter (o.d.) must be less than capillary internal diameter (i.d.). Particularly for capillary of 0.53 mm i.d., standard needles of 0.47mm o.d. and 70 mm length are suitable (e.g., syringe SGE-Chromatec-01-1ul). When using narrow capillary at the beginning of the column, appropriate syringe must be selected.

When using narrow capillary column (0.25 – 0.32 mm), wide bore capillary pre-column of 0.53mm i.d., 0.5 – 2m length can be attached to the front of the main column. The length of pre-column is recommended to choose depending on sample volume, 1m length per each 1 µl of the sample. With wide-bore pre-column standard syringe can be used for injection.

Liner

Instead of usual liners, it's recommended to use liner 6.236.233 from shipping kit, this consists of metal tube and adapter guide. The last provides correct position of the column and needle injection directly into column.

Sample injection

Sample volume is within 0.2 – 2 µl. Injection of small volume should be quick enough so that part of the sample didn't "stick" to needle end.

Inlet temperature

Inlet temperature for injection is set below than solvent boiling point. During analysis, inlet temperature is programmed in the same way as column oven programmed.

Oven temperature

In the beginning of analysis, oven temperature is set below than solvent boiling point, then temperature is programmed.

Gas control

To blow out inlet, vent gas controller should support a few flows 3 … 10 ml/min, carrier gas flow though capillary column is to be set as usually in split mode (see section 1.4.7).

Split, splitless and pulsed pressure injection techniques are similar to the inlet 7.2.

1.5.3 Capillary column connection

Capillary column is installed in the on-column liner up to the stop in the guide sleeve. Needle of the syringe enters the initial section of the capillary column when injecting sample, and the sample is injected directly into the column.

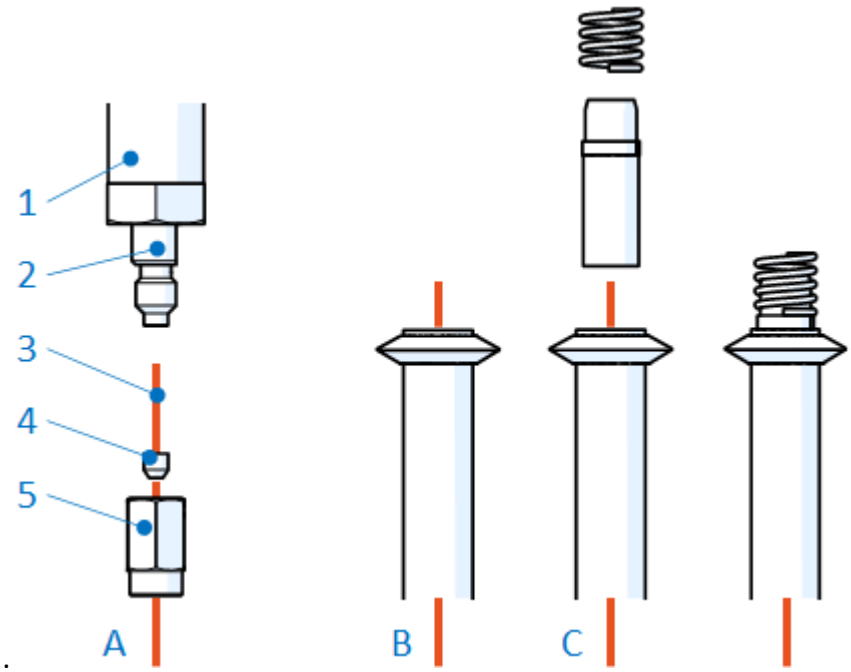


Figure 1.5 –Capillary column connection

Procedure of connecting column to on-column liner:

- Disconnect adapter from the inlet before connecting the column. To do this, unscrew adapter fixing nut, then carefully remove the adapter.
- Using hook from shipping kit or pincers remove liner carefully.

- Check O-ring 6 on the liner. Replace if necessary. Inner diameter of the liner at the ends is slightly different. Liner must be installed in the inlet body with larger internal diameter upwards. Larger inner diameter of the liner is determined during the trial installation of the sleeve 7 in it.
- Put O-ring (8.683.032) on the new liner 10 at 1–2 mm below top edge.
- Insert liner into inlet body. Liner must freely enter into inlet body.
- Put adapter 8 on the handle 5 and turn handle clockwise.

A)

- Put on the nut 5 and the ferrule 4 (e.g. #072626 SGE, with its cone into nut) on the end of column to be installed into inlet.
- Cut column end at (5–20 mm). This prevents column from graphite ingress. The cut should be flat without chips and scratches.
- Wipe cut end of the column with lint-free tissue wetted in alcohol in order to remove dust and other contaminants.

B)

- Insert the capillary column into the inlet fitting in the column oven. Move the capillary column up until it leaves the liner by 5-10 mm.
- Slightly tighten nut 5 in the inlet fitting so that the column is held, but can move up and down.

C)

- Install sleeve 7 (8.230.023) at the end of the column and by pressing sleeve lower it together with the column into the liner.
- Install spring 8 (8.383.068) on the sleeve 7.
- Then tighten the nut by wrench at 60-90 degrees so that column cannot be withdrawn from the fitting (column is tightened by ferrule holder).
- Put on adapter and fixing nut to inlet body. When the nut is screwed on, the adapter pushes the rubber ring on the liner down. At the same time, the liner rests at the bottom of the inlet body.
- Check needle of the syringe freely enters the column.
- Check that there is a carrier gas flows through the column.

Once the column is connected to inlet it should be blown by carrier gas from oxygen at ambient temperature at least for 10-15 min.

1.6 Programmable Split/Splitless inlet 8.1 (P/N 150-1300)

1.6.1 PS/SL inlet 8.1 design

PS/SL inlet 8.1 is intended for injection of samples into capillary column of internal diameter from 0.05 to 0.53mm. The inlet has electronic pneumatic control, see details below. Inlet 8.1 provides 4 techniques of injection into capillary column:

- Split mode;
- Splitless mode;
- Large volume injection with following solvent elimination
- Cold injection with liquid refrigerant cooling. This manual does not cover this feature. The installation of additional equipment (a cryogenic cooling device) required.



Touching inlet top parts, working at temperatures above 300 °C, may cause burns.

Inlet has a lightweight body with heater and fan for quick cooling at programming temperature operation.

Inlet 8.1 has quick-detachable adapter for quick replacement of the liner.

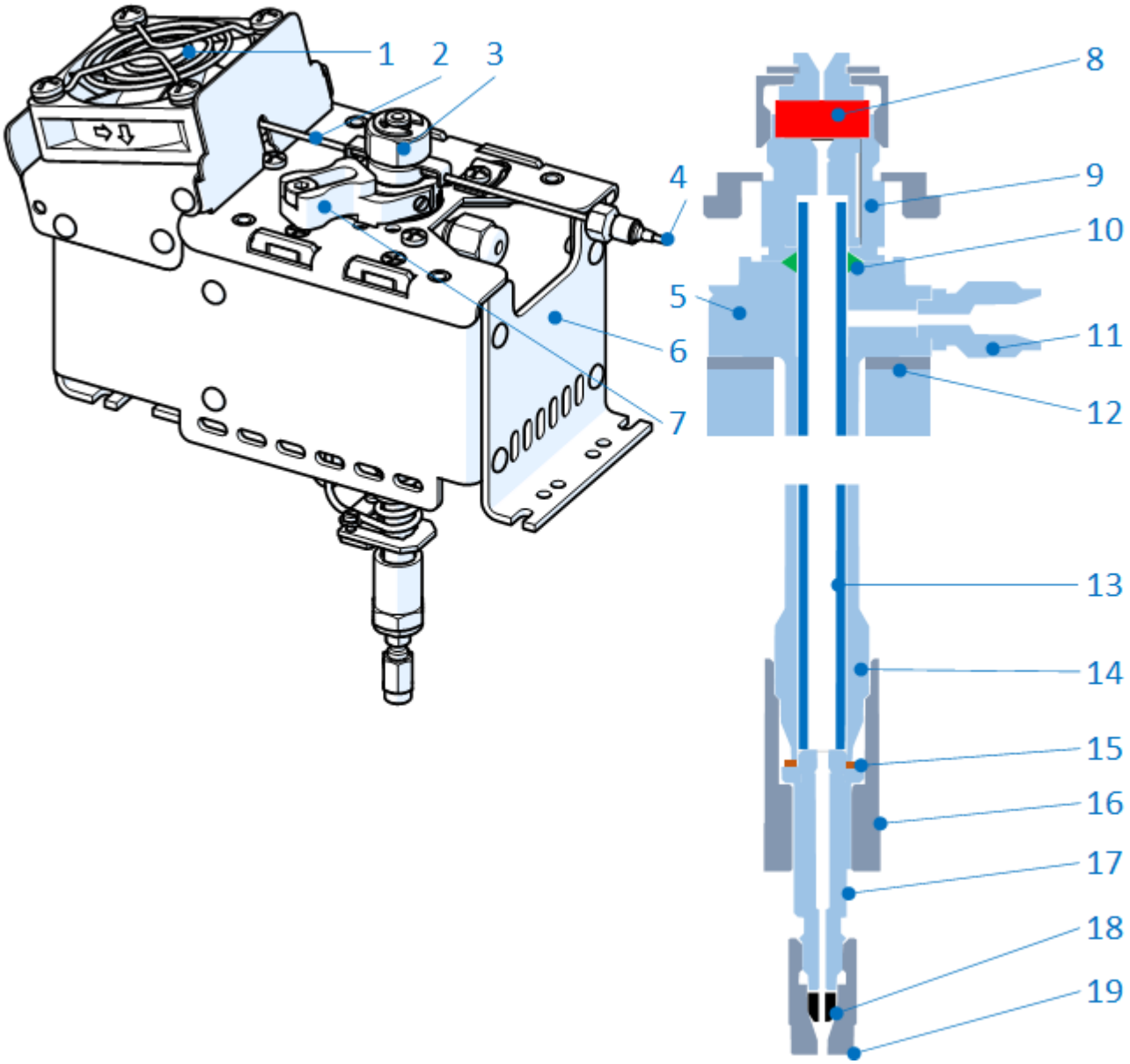
Maximum heating rate from 40 °C to 350 °C is 500 °C/min. Cooling time from 350 °C to 40 °C at 40 °C in the oven is not more than 7 minutes. Cooling fan automatically turns on once “Analysis” state on the GC terminated.

In other things, inlet 8.1 design is similar to SSL Inlet 7.2.

The same liners are applicable for inlet 8.1 as for SSL Inlet 7.2.

Gas connections to inlet 8.1 the same as for SSL Inlet 7.2.

Inlet design is shown below:



1 – fan, 2 – carrier gas tubing, 3 – septum nut (6.482.013-04), , 4 – septum purge tube, , 5 – cover (6.113.097), 6 – cover (6.430.487), 7 – handle (quick access to liner), , 8 – septum, , 9 – adapter (5.889.022-01), 10 – O-ring (8.683.032), 11 – vent fitting, 12 – thermal washer (8.940.007), 13 – liner, 14 – bottom body, 15 – washer (8.940.008), 16 – fitting (8.652.685), 17 – Insert (6.278.006 for liner 99mm length or 6.278.006-01 for liner 113 mm length), 18 – ferrule, 19 – nut (8.930.339).

Figure 1.21 – Inlet 8.1. Design

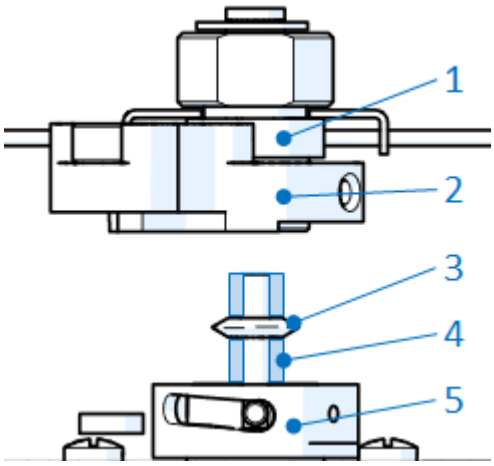
1.6.2 Liner installation (replacement)

It is recommended to replace the liner before starting work with the GC turned off and the inlet and the column oven cooled.



Touching inlet top parts, working at temperatures above 300 °C, may cause burns.

If liner of another length is to be used, change the insert 17 (Figure 1.).



1 – adapter, 2 – handle (quick access to liner), 3 – O-ring, 4 – liner, 5 – inlet body

Figure 1.22 – Liner installation

Liner is to be installed (replaced) as follows:

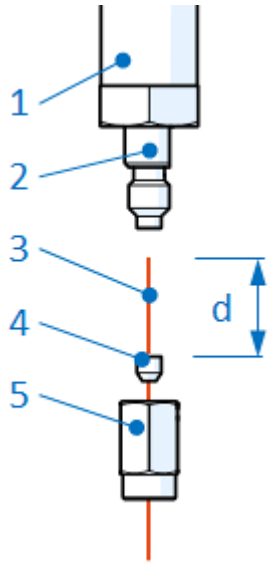
- Disconnect adapter from the inlet. For this rotate handle 2 anticlockwise at 90 degrees, then lift adapter carefully.
- Using hook from shipping kit or pincers remove liner carefully.
- Put O-ring 3 (8.683.032) on the new liner 4 at 1–2 mm below top edge.
- Insert new liner into inlet body down to the stop.
- Install adapter and turn handle clockwise until click.
- After performing the procedure, turn on the chromatograph to transfer the instrumental method, it is recommended to set the temperature of the column oven at room level for 10 to 15 minutes to blow off the oxygen trapped in the inlet
- Check the leakless of inlet in accordance with section 1.7.1.

If liner filled with fiberglass is used, it is recommended to bake it at temperature (up to 270-300 ° C) and a large split (> 1:50) for 30 minutes to remove volatile contaminants. After baking liner, it is necessary to perform blank analysis without injection sample, to make sure there are no false peaks.

It is not recommended to install liner without filling in inlet 8.1, because in this case, the reproducibility of analyses significantly deteriorates.

1.6.3 Capillary column connection

Capillary column is attached into adapter of the inlet 8.1 using suitable ferrule 4 and nut 5 (Figure 1.).



1 – fitting (8.652.685) liner, 2 – Insert (6.278.006 for liner 99mm length or 6.278.006-01 for liner 113 mm length), 3 – capillary column, 4 – ferrule, 5 – capillary column nut (8.930.339)

Figure 1.23 – Capillary column installation

For split/splitless mode, the distance from the bottom edge of the nut to the end of the column (d) is 32 ± 1 mm for a liner of 113mm, 43 ± 1 mm for a liner of 99mm.
For on-column mode, the distance from the bottom edge of the nut to the end of the column (d) is 124.0 – 0.5mm (installation up to the stop, then 0.5 – 1 mm down).
The procedure of column installation into inlet:

- Put on the nut 5 and the ferrule 4 (e.g., #072626 SGE, with its cone into nut) on the end of column to be installed into inlet.
- Cut column end at (5–20 mm). This prevents column from graphite ingress. The cut should be flat without chips and scratches.
- Wipe cut end of the column with lint-free tissue wetted in alcohol in order to remove dust and other contaminants;
- Locate nut and ferrule 5 at required distance (d) from the column end. For convenience, use column measuring tool from shipping kit. Install column end into measuring tool, align column end at mark (1) for 113mm liner or (2) for 99 mm liner, tighten the nut by wrench at 60-90 degrees so that column cannot be withdrawn from the fitting. Then release nut.
- Insert the column into inlet adapter, tighten nut by hand.
- Then tighten the nut by wrench at 45-60 degrees so that column cannot be withdrawn from the fitting (column is tightened by ferrule holder).

Once the column is connected to inlet it should be blown by carrier gas from oxygen at ambient temperature at least for 10-15 min.

1.6.4 Large volume injection (LVI) operating parameters

This mode allows injecting large volume of the sample (up to 50 µl) in order to increase sensitivity at determination of trace level components. Sample is injected into cold inlet so that solvent is evaporated steadily and blown out through the vent line, analytes are focused in liner. Then vent channel gets closed, inlet is heated quickly, analytes are transferred by carrier gas from the inlet into GC column. Due to preliminary elimination of the solvent, column overloading doesn't occur, components focused in the liner and then exposed to quick heating are introduced into column in a narrow bandwidth.

When using large volume injection with following solvent elimination, the solvent should be selected so that its boiling point was at least 100 °C below than boiling point of the most volatile compound.

Recommendations on large volume injection are suggested below

Apparatus

Inlet should provide high heating rate up to 500 °C/min.

Syringes are selected depending on volume of sample to be injected. Multiple injection using syringe of smaller volume is possible.

Liner. When using large volume injection with following solvent venting, it's recommended to use liner splitless (# 20860 Restek or # 0920831 SGE) filled with glass wool. For better trapping of volatile organic compounds while solvent venting, small section in the liner may be filled with suitable sorbent (e.g., Tenax or Carbopack), however take attention this may result to poor desorption of heavier components.

Inlet temperature

While injecting inlet temperature is set at 5 – 10 °C below than solvent boiling point. After injection inlet temperature is heated with rate 200 – 500 °C/min. Final temperature level depends on analytes boiling points. When using sorbent in the liner, inlet maximum temperature must not exceed sorbent operating limit by temperature. Inlet is to be kept at maximum temperature within 2 – 5 minutes so that analytes are transferred into column, then temperature can be decreased.

Oven temperature

Initial column oven temperature usually is the same as inlet temperature, though it may vary ±10 °C. Oven programming start is set as soon as inlet reached its maximum temperature.

Gas control

While sample injection and solvent elimination takes place, split vent flow is from 50 to 200 ml/min, then it is set to 0 ml/min with inlet heating start. Once heating is terminated, vent flow rate is set at low level 10 – 30 ml/min.

Solvent vent time

Time for solvent elimination depends on solvent boiling point, liner filling type, inlet temperature, vent flow rate. Usually it is within 2 – 15 seconds. Solvent vent time should be set so that the most volatile analyzed compounds were not lost.

Diagrams demonstrating gas control, oven and inlet temperatures at large volume injection mode are described below (Figure 1.14).

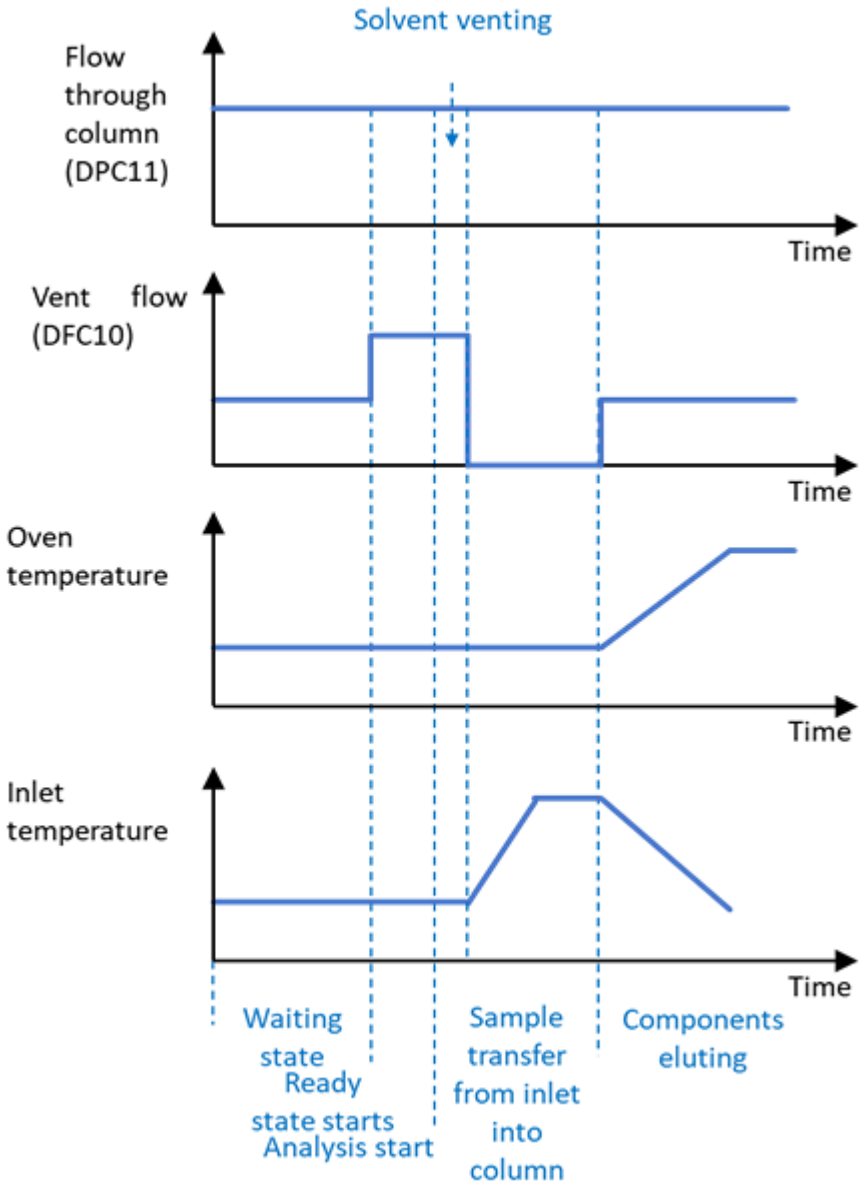


Figure 1.14 – Gas control, inlet and oven temperatures at LVI mode

Injection procedure

- If the GC is on the state "Waiting" press START to transfer into "Ready" state.
- Once "Ready" state occurs, quite massive flow rate is set through vent channel to eliminate solvent.
- Inject sample. Sampling of large volume should be carried out slowly, for 3 – 10 sec. Multiple injection is possible, for example, 30 µl injection can be made with 10 µl syringe – 3 times by 10 µl.
- Press START once last injection is done.

- After solvent venting (2-15 sec), vent channel shuts off, the rapid heating of the inlet starts, at heating target components are transferred from the inlet into column similarly to splitless mode.

1.7 Inlet maintenance

Before inlet maintenance the oven should be cooled down to temperature preventing chromatographic column from failure or its performance deterioration.



Inlet parts can be heated to temperature high enough to cause burns.

All the procedures involving disassembling or assembling of inlet are to be performed at its temperature got down below 50 °C.

Inlet maintenance is recommended to perform in the beginning of workday at GC switched off.

If some maintenance procedures are to be performed at the GC being turned on set null pressure value to pressure gas controllers connected to the inlet, in order to avoid carrier gas loss.

1.7.1 Inlet checks for leaks

Inlet check for leaks is to be carried out after change or regeneration vent trap, change of O-ring, liner, GC column, as well as other disconnections of pneumatics in the inlet system.

Check for leaks of capillary inlet is convenient to monitor by difference between flow rates of input gas controller (DPC11) and output vent gas controller (DFC10). On the state “Ready” at the same conditions (GC mode, the same GC column), the difference must be constant. Increased gap indicates to leak.

In daily operation, leak usually arises through inlet septum worn out.

Automatic leak check can be adjusted in CONFIG key menu, switch on “Leak check” option and specify allowable “Leak value”, see section 3.7.6-part 1 UM for details.

It is convenient to control leakless of the **capillary inlet** by comparing the difference in the flow readings of the input (DPC11) and output (DFC10) controllers. At the "Ready" state under constant conditions (chromatograph mode, installed column), the difference must be constant an increase difference of value indicates the appearance of a leak.

To check leakless of the packed inlet, remove the column, install the plug 8.632.195 from the spare parts kit instead. Set the working or increased carrier gas flow rate to 50 ml/min on the DFC to inlet. After 5 minutes, make sure that there is no flow through the DFC.

General recommendations to check for leaks are described in section 3.4.4., part 1 UM.

1.7.2 Septum change

Inlet septum state is one of the most critical factors for obtaining satisfied chromatographic analysis result. Recommendations on septum usage are specified in section 1.2.3.

Septum lifetime depends on syringe needle diameter, needle tip and other factors, lifetime is usually within 100 – 200 injections. In addition, there is a great dependence on the state of the needle end, which should not have "cutting" edges.

Septum is recommended to change in the beginning of workday at the GC turned off. Septum change procedure:

- Unscrew inlet top nut. Wrench 8.392.006 could be used for unscrewing (see shipping kit).
- Remove the worn septum.
- Insert the new septum.
- Screw the nut at 180 – 270 degrees from the point of contact septum to nut insert.
- Tighten radiator nut until the backlash in the threaded connection is eliminated.
- Pre–tighten the nut by turning it clockwise at an angle from 180 to 270 degrees, while significant deformation of the septum material (silicone rubber) occurs; observing these conditions, the connection of the adapter – septum pair can be considered sealed.
- To adjust the tightening force of the nut and check the tightness, turn on the chromatograph, transfer the operating mode, it is recommended to set the temperature of the column oven at room level for 10 to 15 minutes to blow off the oxygen trapped in the inlet after replacing the septum.

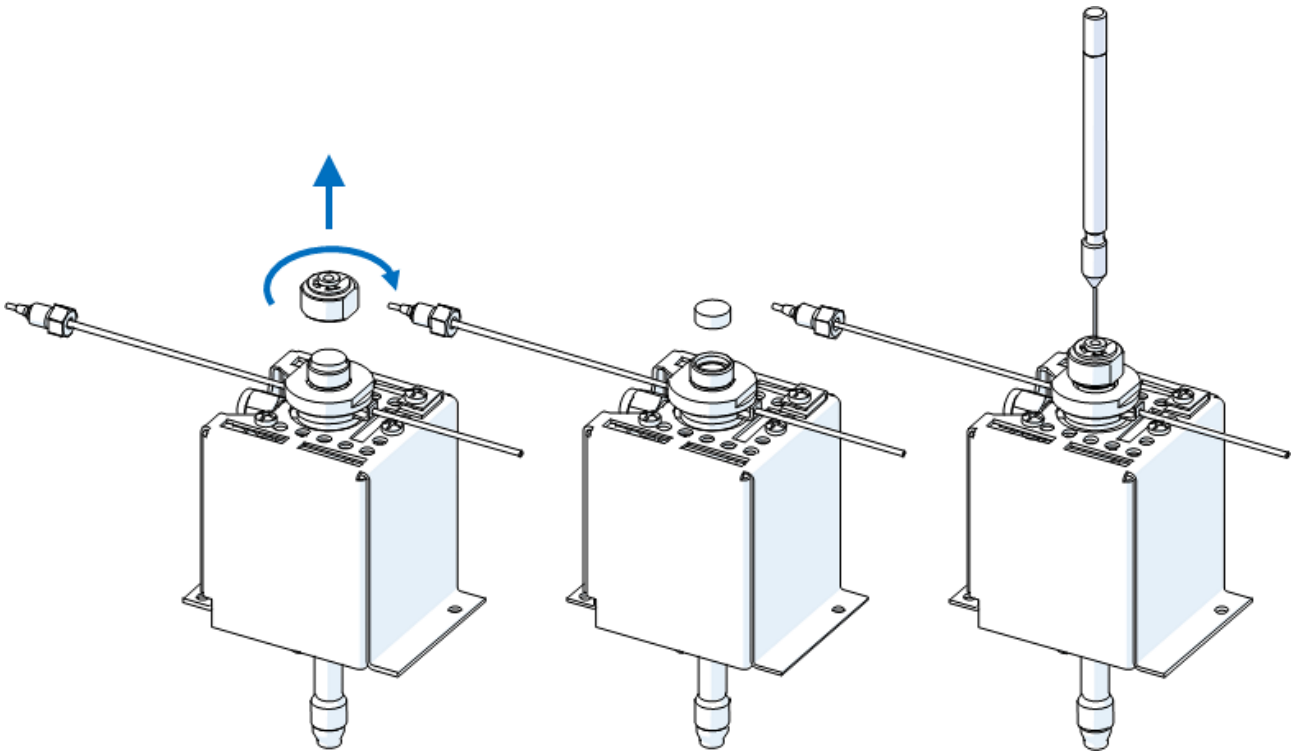


Figure 1.6 – Septum change

- For a capillary inlet, set the parameters of the carrier gas (pressure, velocity or flow) and vent gas, and test the inlet path by observing on the chromatograph display the measured values of the carrier gas and gas flow along the vent line. Quantitative of the difference in these costs is taken as the state of tightness of the inlet path with an intact septum.
- Gradually loosen the nut until there is a backlash in the threaded connection, then screw it onto the inlet until the backlash in the threaded connection is eliminated; pre-tighten the nut by turning it clockwise at an angle from 60 to 90 degrees, while the septum material is slightly deformed.
- Perform a 2-3-fold puncture of the septum with a needle 6.054.005 (from the spare parts kit) oriented along the axis of the inlet. When puncturing, pay attention to the smoothness and ease of passing the needle. If the needle passes with effort, this indicates a stretched septum, when inserted with a micro-syringe, the needle may be damaged.
- To test the inlet path, fixing the flow rates of the input carrier gas and vent flow; the invariance of quantitative value of the difference in these costs indicates the tightness of the inlet path with a punctured septum, an increase in it indicates the leakiness of the septum, i.e., the appearance of additional gas flow through it.
- By slightly turning the nut, fix its position in such a way that the tightness of the tract is maintained with minimal tightening of the nut.

While working, permanently monitor inlet leak-free condition (in each 30 – 40 injections, as described in previous section.

If leak revealed, it can be eliminated by screwing nut clockwise at about 30°.

This way of leak elimination can be applied up until nut didn’t screw to the stop.

Typical septum lifetime is not less than 200 injections at following above said recommendations.

At excessive tightening septum nut or/and using bended needle, septum lifetime may reduce to as low as 5 injections.

In order to avoid breakage of the needle of the micro-syringe and reduce the life of inlet septum, tighten the nut with minimal effort, ensuring the tightness of the inlet path. The passage of the needle through the membrane should be easy, but not violating the tightness of the inlet.

The life of the inlet septum can be increased if, when replacing the liner, the adapter with the nut installed on it is removed (only the lower adapter mounting nut is unscrewed), since this operation does not violate the tightness adjustment of the adapter – septum pair.

When using a septum with a diameter of 11 mm instead of a septum with a diameter of 10 mm, the frequency of tightening the nut can be increased to 150 injections, which will further increase the total number of punctures with a syringe needle without losing the tightness of the inlet.

1.7.3 Sealing O-ring and liner replacement

It's recommended to check O-ring state periodically. Higher the operating temperature of the inlet, the faster the liner ring wears out.

It is recommended to replace the liner and sealing O-ring before starting work with the device turned off.

If O-ring became hard and looks flattened, it needs to be replaced (Figure 1.10):

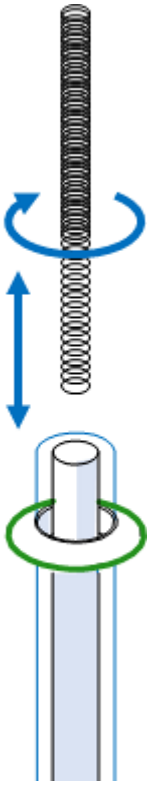
- Unscrew adapter nut. Use wrench 8.392.006 from shipping kit.
- Divert directly upwards adapter assembly in order to avoid liner top edge chipping.
- It's important, resign O-ring having been heated may stick to glass liner and to metal surface of inlet body, adapters assembly. In this case separate stuck O-ring from inlet body using hook 6.054.001.
- Grab O-ring with pincers and pull-out O-ring together with liner upward.
- Install new ring on the distance (1–2) mm from liner top edge and assemble the liner.

When tightening inlet adapter nut and the septum nut, do not apply excessive force. Usually, the effort of tightening by hand is enough.

Liner cleanness has existent influence on analysis quality. If liner contaminated with septum scraps or sample remnants, there could arise ghost peaks on chromatogram.

Operations with the liner are recommended to be carried out with gloves.

Liner clearing:



- Procedure for installing (replacing) the liner, depending on the type of inlet, is described in sections 1.3.2, 1.4.3, 1.6.2.
- Examine liner. If there are any contaminants, clean inside surface of the liner with spring 7.067.011 and lint-free tissue wetted in suitable solvent. Solvent could be chosen depending on sample nature, but not aggressive to inlet material;
- It is recommended to remove the contaminated glass wool (fiberglass) and install a new glass wool or fiberglass instead. Recommendations on the choice of liner and the choice of filling are given in section 0.
- Assemble inlet.

2 GAS VALVES

In the "Control Panel" of the Chromatec Analytic software, the valve is labeled as Valves.

The configuration of valves and supply gases is configured in accordance with Section 3.2.1 Sample input ports, User Manual 214.00045-51I, Chromatec Analytic Software. Setting the parameters of valves and carrier gases in the analysis mode is performed in accordance with the section Sample input ports, User Manual 214.00045-51I, Chromatec Analytic Software.

2.1 Overview

Valves are intended for gas sampling (gas sampling valves), switching of columns or carrier gas streams in GC (gas switching valves).

Rotary valves manufactured by Chromatec are described in this section. Piston injector, being a valve of piston type, is described in User Manual 214.4.464.020UM, supplied as an option (by order).

Valves of 3rd party manufacturers are described in corresponding documents accompanied with devices (option).

2.2 General technical specifications

Table 2.1 – Valves technical specifications

Parameter	**Value**
Number of ports (depending on valve modification)	4; 6; 10
Ports internal diameter, mm	1.0
Flow capacity1 (at nitrogen pressure 0.045 MPa), ml/min, not less	200*
Maximum temperature, °C	150
Maximum gas level, kPa	600
Fitting connection	M6x0.75 female, 1.6mm tubes
Materials, of path flow	Stainless steel or aluminum, PTFE, glass

Technical specifications for valves of 3rd party manufacturers (e.g., VICI) are specified in accompanied documents.

*Flow capacity of valve ports is measured without trap 5.884.098.

When using Chromatec made valve as a sampling valve in combination with small volume loops, volume of valve internal ports shall be taken into account, which is about 20 µl.

2.3 Description and operation

2.3.1 Introduction

In chromatograph according to gas diagram, applied the valves which may vary in:

- number and inside diameter of ports (4, 6 or 10 port; 1.6mm or 3mm ports diameter);
- automation (manual or automatic);
- heating option (non-heated or temperature-controlled);

On default valves with ports for tubing 1.6 mm and fitting thread M6x0.075 female is suggested (nuts and ferrules included). Valves for a 3mm tube (thread M8x1 inside) are available on request for old chromatographs.

Delivery set

Valves are supplied with ferrules and nuts. Sample loops are not included in the set at delivery (option).

When ordering valves and sample loops, specify catalogue number according to valve catalogue 214.4.061.050-01KDS, this contains full information about valve and sample configuration.

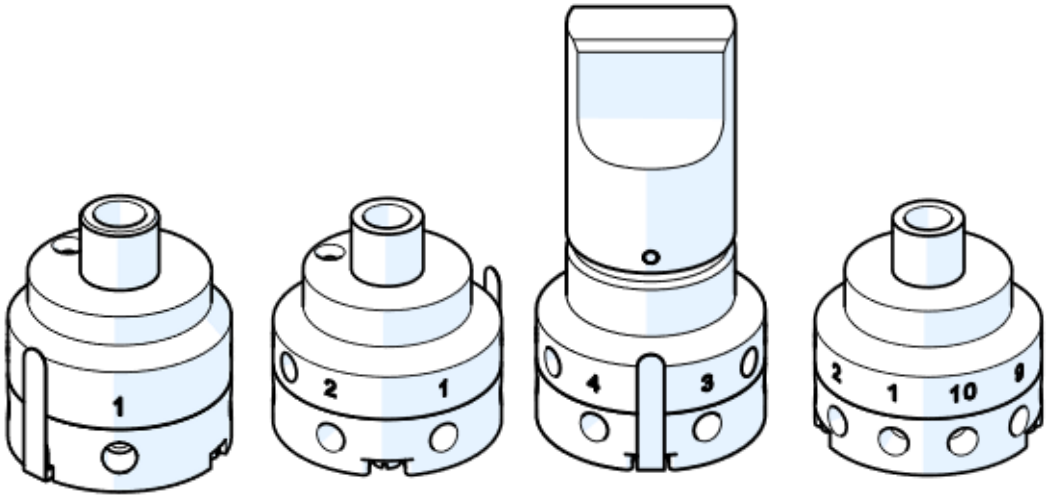
A tag connected to valve body indicates serial number and year of manufacturing.

Pneumatic and electrical connections

Automatic valves are controlled by electrical actuator. Cables for electrical actuator, heater and temperature sensor are connected to appropriate slots in power board according to flow diagram marking (see section 3.7, part 1 UM).

Valves mounted in particular GC according to order specification and flow diagram are listed in flow diagram specification.

Table 2.2 – Rotary valves. Descriptions

Overview				
Number of ports/positions	4 ports 2-positional	6 ports 2-positional	6 ports (3-positional)	10 ports 2-positional
Body material	Stainless steel	Stainless steel Aluminum	Stainless steel	Stainless steel Aluminum
Body material	5.885.002	4.464.024-01.03 4.464.024-01.04	4.464.024-01.05	4.464.025-01.03 4.464.025-01.04

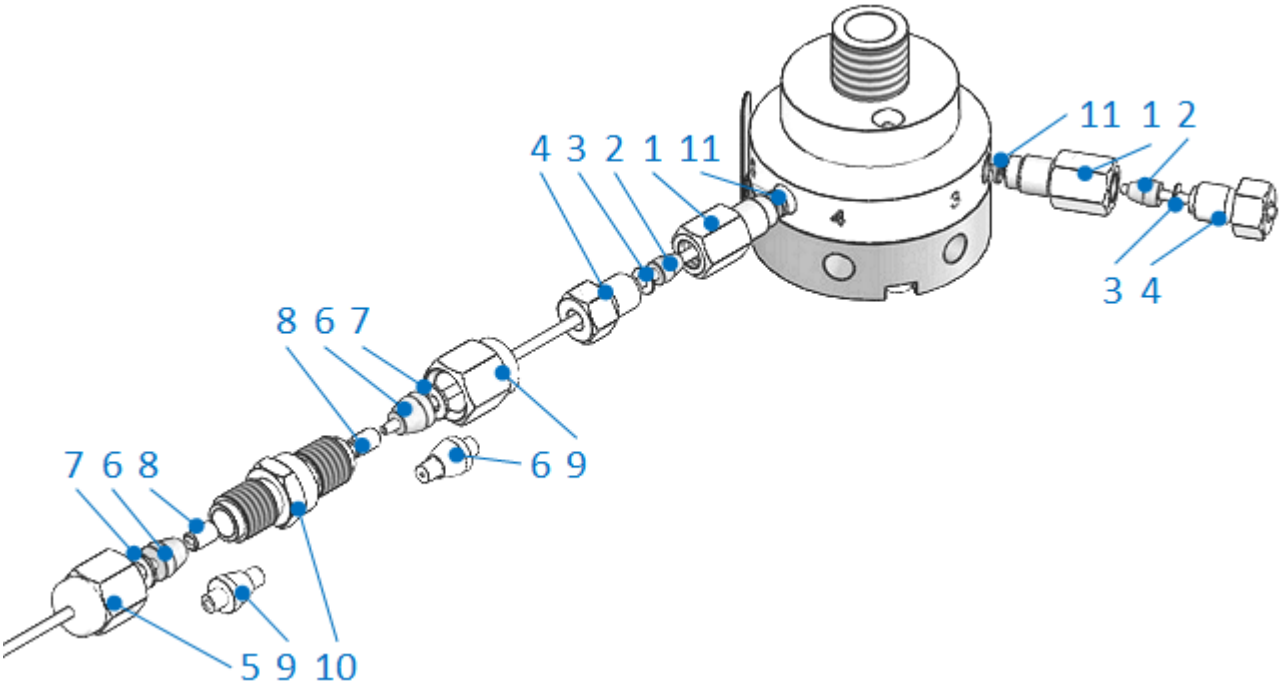
Valves may operate as a sampling and/or switching valves, depending on particular application, have various flow connection diagrams. Flow diagram of valves in particular GC is given in part 3 UM, attachment B (flow diagram).

To connect tubes 1.6mm in valve ports, nuts 8.930.161 and ferrules 8.658.039-01 (stainless steel) or 8.658.052 (rubber) are used. Rubber ferrules are used at valve temperatures no higher than 50°C, provided there is no influence of air components on the measurement results, in other cases stainless steel ferrules are used.

Recommendations for the correct sealing of the tubes are given in section 3.4.4. UM – Installation Instructions.

Purge housing

All Chromatec made valves have additional fittings for purge housing in its construction. This allows reducing influence of ambient components diffusion on analysis results (especially important when trace levels of ambient components are to be determined). Purge housing connection is provided by tubing 1.6mm and flow restrictor 5.150.015-02 from carrier gas connector (400 kPa) (Figure 2.1).



1 – fitting 8.652.494**; 2 –** ferrule 8.658.053**; 3 –** washer 2**; 4 –** nut 8.930.161**; 5 –** nut 8.930.167**; 6 –** ferrule 8.658.052**; 7 –** insert 8.223.429**; 8 –** sleeve 8.220.231**; 9 –** ferrule 8.658.024**; 10 –** flow restrictor 5.150.015-02**; 11 –** O-ring 8.685.076.

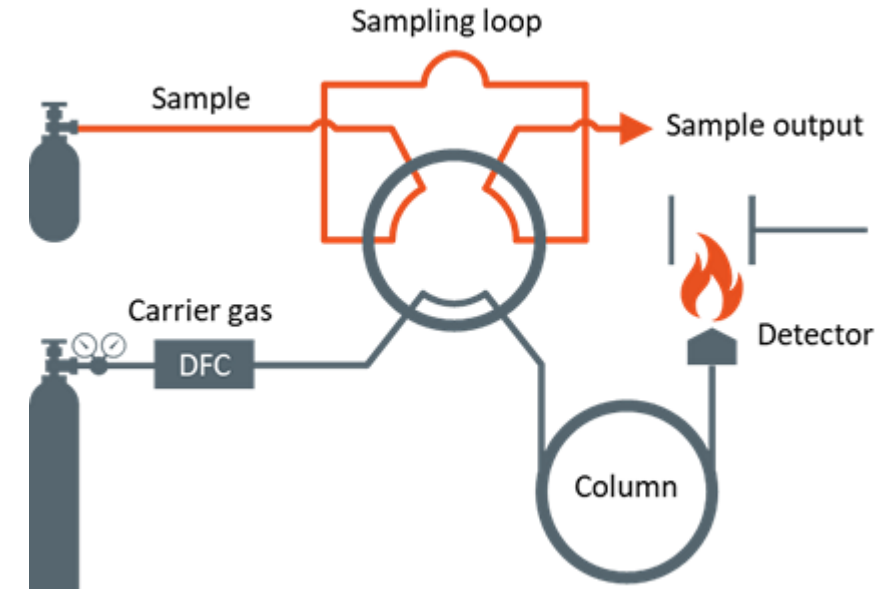
Figure 2.1 – Purge housing connection 214.4.464.024-01.03(.04) and 214.4.464.025-01.03(.04)

2.3.2 6-port valve (2-positional)

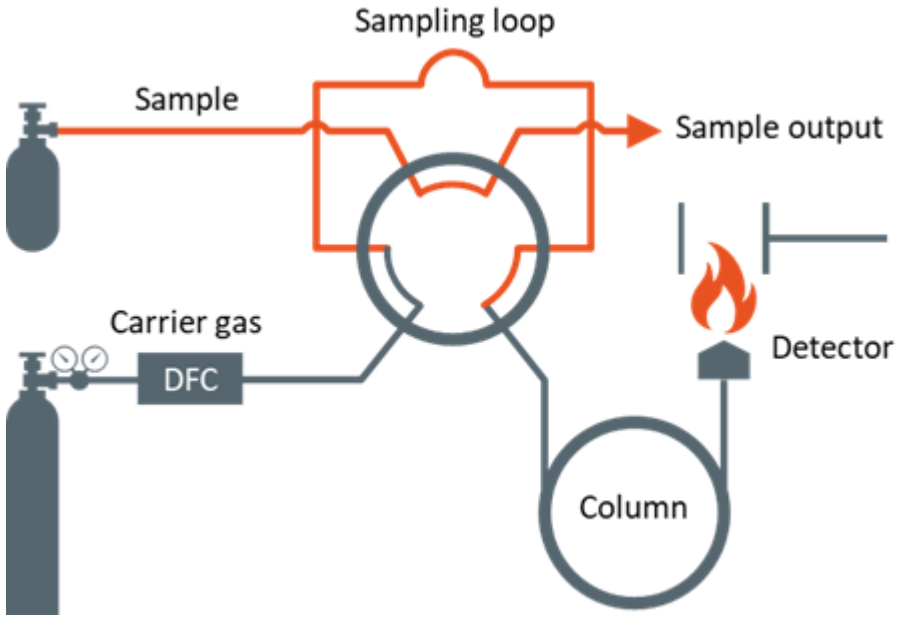
Connecting valve with gas sample source it is important to reduce the total internal volume of sampling line as more as possible and avoid dead volume occurrence in connections.

Operation diagram of 2-positional 6-port gas sampling valve is shown below (Figure 2.2). In valve position “Sampling” (position A) sample flow is blown through a sample loop (D1), carrier gas flow passes directly into chromatographic column. The arrow shows handle position.

When rotating to position “Injection” (clockwise) sample loop is included into carrier gas flow and sample in loop is carried by gas flow into chromatographic column.



Sampling position



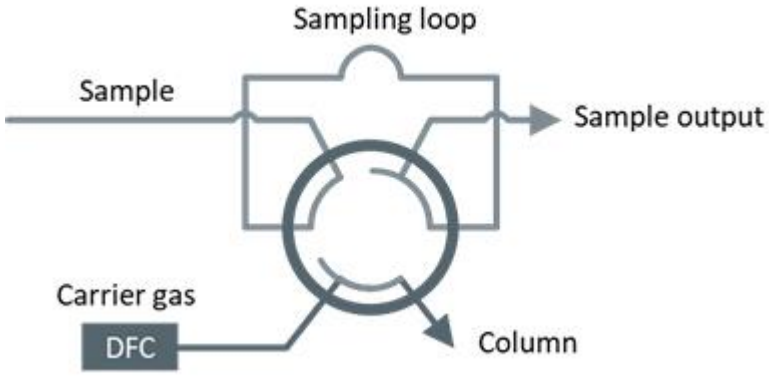
Injection position

Figure 2.2 – 6-port valve operation diagram

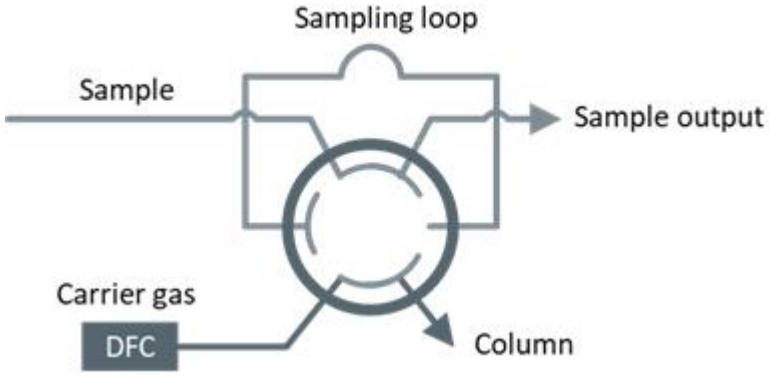
Gas lines connections. Gas lines connections diagram for valve operation in GC is shown on – Figure 2.2. If necessary, perform (check) recommended connections. If there could be solid contaminants in sample flow, it's recommended to fit a filter 5.884.098 containing 5µm frit to fitting "6" (Sample inlet). Fitting "5" (if necessary) is to be connected for utilization. Make check for leaks (see section 3.4.4, part 1 UM).

2.3.3 6-port valve (3-positional)

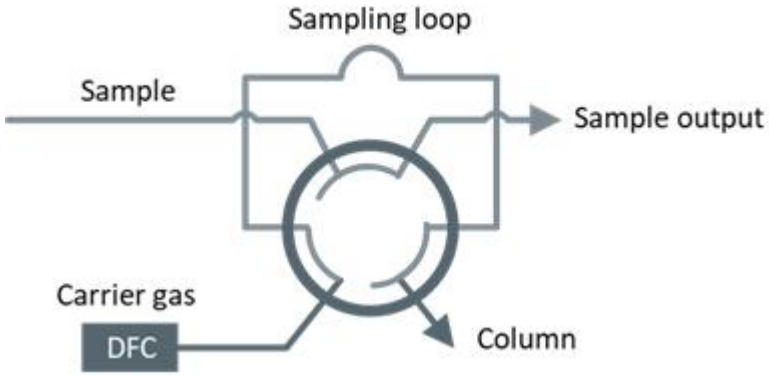
Operation diagram of 3-positional sampling valve is shown below:



Left-side position “Sampling”



Middle position “Sample is locked”



Right-side position “Injection”

Figure 2.3 – Operation diagram of 3-pos. valve

In valve position “Sampling” (the outside left position of the handle, anticlockwise) sample flow is blown through the sample loop (focusing trap), carrier gas flows directly into chromatographic column. Handle is “outpressed”. In this position sample passes through the sample loop immersed

in cooling agent (liquid nitrogen or carbon dioxide), thus heavy analytes are trapped and concentrated in the loop).

After sampling and focusing of analytes in the cooled sample loop, valve is to be rotated to the middle position. Handle is “outpressed”.

In this position loop is locked (sample closed), carrier gas goes flowing through the valve into column. Sample loop is to be pulled out from the cooling agents' vessel for heating.

Once sample loop has been heated up to required temperature level, valve handle is to be rotated to “Injection” position. To rotate to “Injection” position, the handle is to be “pressed” first, and then rotated clockwise.

In “Injection” position, sample loop gets into carrier gas flow and heated analytes are carried out into chromatographic column.

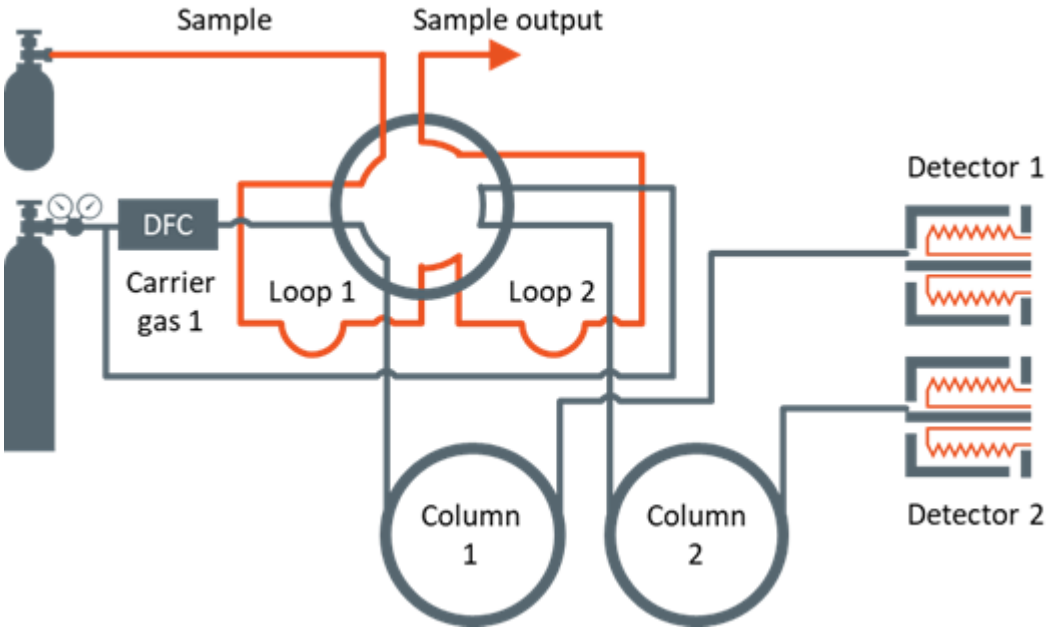
Gas lines connection. Gas lines connections diagram for valve operation in GC is shown on. If necessary, perform (check) recommended connections. If there could be solid contaminants in sample flow, it’s recommended to to fit a filter 5.884.098 containing 5µm frit to fitting “6” (Sample inlet). Fitting “5” (if necessary) is to be connected for utilization. Make check for leaks (see section 3.4.4, part 1 UM).

2.3.4 10-port valve

Operation diagram of the valve is shown below.

In valve position “Sampling” sample flow is blown through the sample loops (D1, D2), carrier gas flows pass directly in chromatographic column. The arrow shows valve handle position.

At valve handle turning to position “Injection” (clockwise) sample loops are included in appropriate carrier gas flow and sample in loops are carried by gas flows into chromatographic columns.



Sampling position

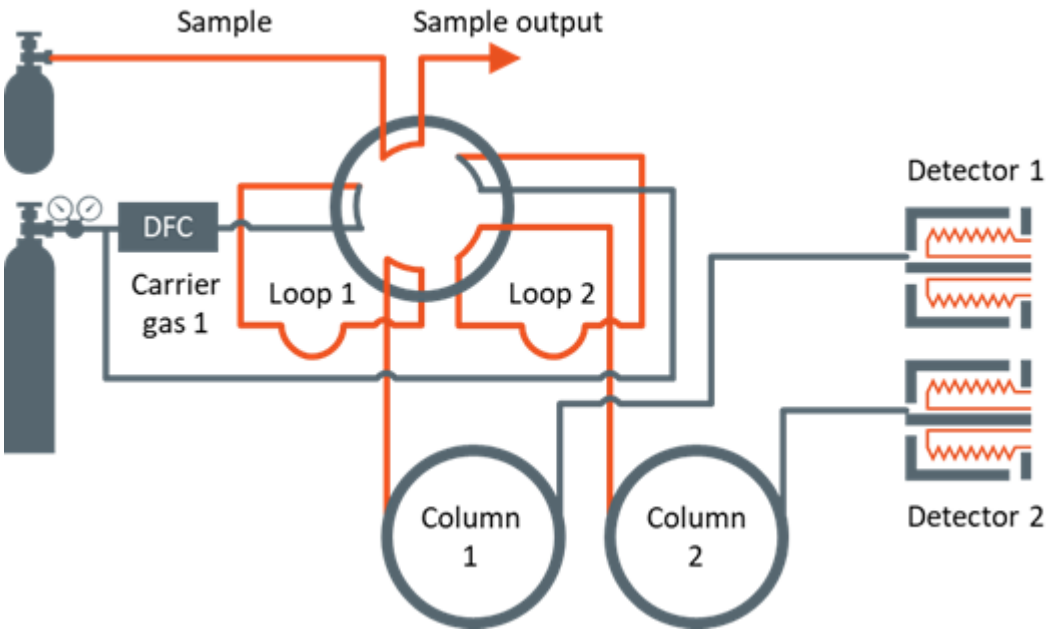
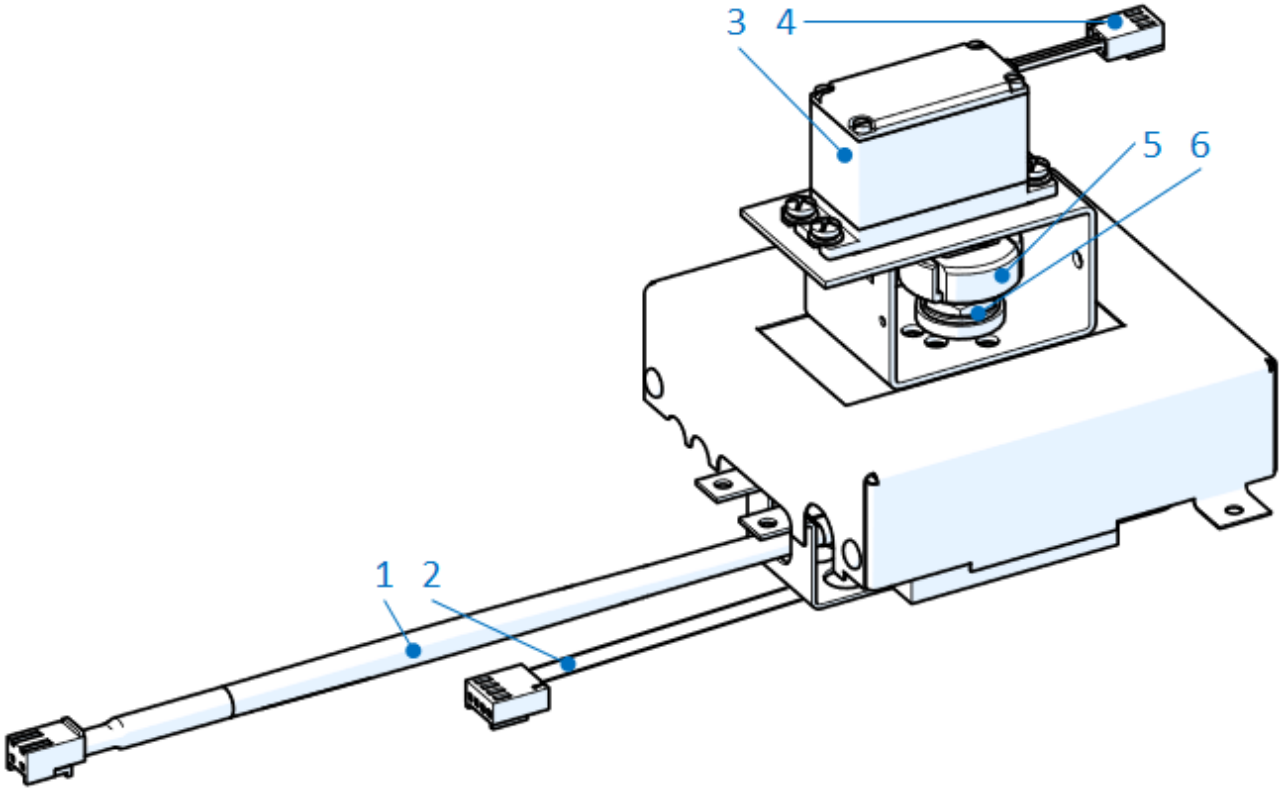


Figure 2.4 – 10-port valve operation diagram

Gas lines connection. Gas lines connections diagram for valve operation in GC is shown on. If necessary, perform (check) recommended connections. If there could be solid contaminants in sample flow, it's recommended to fit a filter 5.884.098 containing 5µm frit to fitting "1" (Sample inlet). Fitting "2" (if necessary) is to be connected for utilization. Make check for leaks (see section 3.4.4, part 1 UM).

2.3.5 Automatic / temperature-controlled valves

Automatic temperature-controlled valve overview is below – Figure 2.5. Electrical actuator, being fitted on the bracket by screws, is connected to valve by means of shaft and bushing. The valve is joined to the bracket by nut. A pin prevents valve body from rotating relatively to the bracket and actuator.



1 – Heater cable, 2 Temperature sensor cable, 3 – Actuator, 4 – Actuator cable, 5 – Connecting elements (shaft and bushing),6 -Nut

Figure 2.5 – Rotary valve. Assembly with actuator and heating panel

Automating and heating functions of the valve are controlled by means of power board. Cables of actuator, temperature sensor and heater are connected to appropriate slots in the power board. Detailed information is described in section 3.7, part 1 UM. Extending cable 6.644.331 is to be used to connect actuators cable.

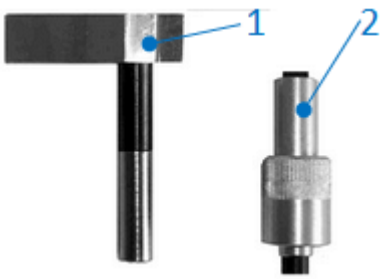
Valves in the GC are mounted on the GC platform or on the left side bracket. Depending on particular location heating panel 5.863.167 or 5.863.193 is used respectively (see details in section 3.3, part 1 UM).

Actuator disassembling

If one need to disassemble actuator from automatic valve and use it as a manual valve, follow procedure below:

- Remove 4 screws fitting actuator;
- Remove actuator and connecting elements (shaft and bushing);
- Unscrew nut with the wrench (14mm size), remove the nut and the bracket;
- Connect extender 6.306.023 and handle 6.465.001 to operate manual valve.

To locate handle outside of GC cover, there are holes in the covers.



1 – Handle 8.465.001, 2 – extender 6.306.023

Figure 2.1 – Manual switching elements

2.4 Gas connections

Gas controllers are connected to valve in the same way as to inlets.

When operating with packed columns, gas controller DFC-10 (Figure 1.4) or DPC-11) is attached at the valve inlet carrier gas supply.

When operating with capillary columns, input flow is formed by pressure gas controller (DPC-11. A2), vent flow is formed by flow gas controller DFC-10.

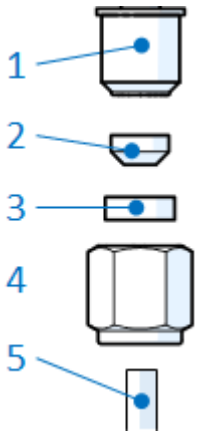
2.5 Columns connection

Packed column connection depending on column type and ferrules material is shown below.

Rubber ferrules are recommended for use at temperature in the oven less than 250°C.

Assemble nut 8.930.172, insert 8.223.013 and rubber ferrule 8.220.380 onto the packed column end.

Insert the end of the column into valve's fitting up to the stop. Hold the column in this position and tighten the nut finger tight.



1 – Fitting, 2 – ferrule 8.220.380 (rubber), 3 – insert 8.223.013, 4 – nut 8.930.172, 5 – packed column

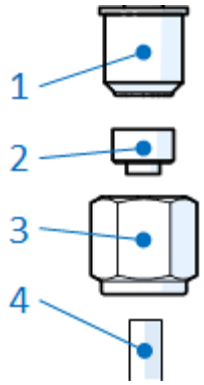
Figure 2.7 – Packed column connection using rubbery ferrule

Graphite ferrules are allowed to use in the oven up to 450°C.

Assemble nut 8.930.172, ferrule holder 6.453.038 onto the packed column end.

Insert the end of the column into valve's fitting up to the stop. Hold the column in this position and tighten the nut finger tight. Use a wrench to tighten the nut an additional 30-60 degrees.

Be careful when tightening glass column with graphite ferrules in order to avoid its breakage.



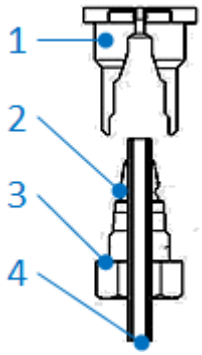
1 – Fitting, 2 – ferrule 8.220.380 (rubber), 3 – insert 8.223.013, 4 – nut 8.930.172, 5 – packed column

Figure 2.8 – Packed column connection using graphite ferrule

To connect column of type 3 appropriate fitting should be installed in the oven.

To connect column of 3mm outer diameter ferrules 8.658.038-01 are to be used, for column of 3.18mm (1/8") o.d. use ferrules 8.658.038-03.

Insert the end of the column into detector's fitting up to the stop. Hold the column in this position and tighten the nut finger tight. Use a wrench to tighten the nut an additional 30-60 degrees.



1 – Fitting, 2 – ferrule, 3 – nut 8.930.114, 4 – column

Figure 2.6 – Packed column (type 3) connection using metal ferrule

Capillary column connection

To connect the capillary column to sampling valve, a tee 6.453.131-03 is used (4, Figure 2.10), which performs the function of a flow splitter when injecting sample into the column.

Nut 3 (8.930.172) and ferrule 2 (6.453.038) are installed sequentially on tee 4, as shown in the figure. Insert T-branch fitting into fitting up to the stop, and sealed by nut 3. Effort of nut tightening "by hand", with a subsequent 180-270-degree rotation for a new ferrule and 45-60 degrees for repeated sealsings.

The side tube 10 with a diameter of 1.6 mm with a ferrule 8 (8.658.039-01) and a nut 9 (8.930.161) is connected to the vent line.

Column connection:

Put nut 6 (8.930.099) and ferrule 5 (for example, #072626 SGE, cone down) on the capillary column.

Cut column end at (5–20 mm). This prevents column from graphite ingress. The cut should be flat without chips and scratches. Wipe cut end of the column with lint-free tissue wetted in alcohol in order to remove dust and other contaminants;

Locate nut and ferrule 5 at a distance of at least 30 – 40 mm from the bottom edge of the nut to the end of the column (above the level of T-branch fitting), for convenience, the position of the column under the nut can be marked with a marker or a piece of rubber (septum) can be installed.

Insert the column into the tee, tighten the nut 6 by force "by hand", align the column level. Holding the tee 4 from turning with the key (by 8), turn the nut 6 with the key (by 7) by 60-90 degrees so that the column cannot be pulled out of the nut (the column is fixed with a ferrule).

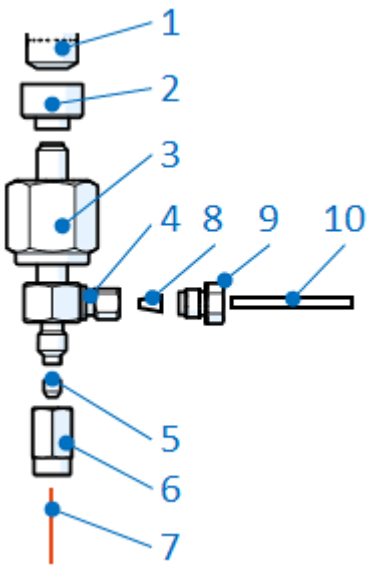


Figure 2.10 – Capillary column connection

2.6 Sampling valve operation

Operation procedure with gas sampling valve is described below.

- Before operation beginning, verify that valve position is “Sampling”, chromatograph should be at status “Ready”.
- Open on/off valve on the sample source.
- Pass sample flow through the sampling line. The volume should be 10-20-fold of the total volume of sampling line and valve's loop is.
- Close on/off valve on the sample source.
- Pressure in loop should be balanced with atmospheric (pause should be carried out for 10 seconds after sample source was closed).
- Do transfer valve to “Injection” position by turning valve handle clockwise and press START on chromatograph control panel.
- When analysis is finished the valve should be turned into position “Sampling”.

Automatic valve operation is carried out in the same way. Valve rotating into analysis position is provided automatically at START button press.

2.7 Valve maintenance

Except from possible leaks (overflow) elimination, customer is not recommended to repair any defects on one's own. If defects revealed, please contact manufacturer or regional representative.

2.7.1 Sample loop replacement

To replace sample loop in the sampling valve, follow procedure:

- Unscrew nuts connecting sample loop to the valve and remove the loop.
- Install nuts 8.930.161 and ferrules 8.658.039-01 on the new loops ends.
- Carefully install loops ends into the same valve fittings up to the stop and screw nuts.
- Check connections for leaks (see section. 3.4.4, part 1 UM).

2.7.2 Check for leaks

Check for leaks of valve ports and gas lines connected to the valve may be carried out either:

- Without gas lines dismounting, check for leaks the whole pneumatic chain;
- With gas lines dismounting, by consequent excluding of pneumatic chain sections.

Check for leaks is carried out using digital flow and pressure controllers or by soaping. General recommendations on checking for leaks are described in section 3.4.4., part 1 UM.

2.7.3 Check for "overflow" between ports in sampling valve

Check for "overflow" between carrier gas ports and sampling ports in sampling gas valves is important. For this purpose, it is necessary to create in gas lines excessive pressure (0,4 – 0,64) MPa. Input of sampling line is to be sealed, gas line of sample output to lower in distilled water. Gas bobbles appearance is not allowed.

After check termination the rest of water should be dried by lint-free tissue.

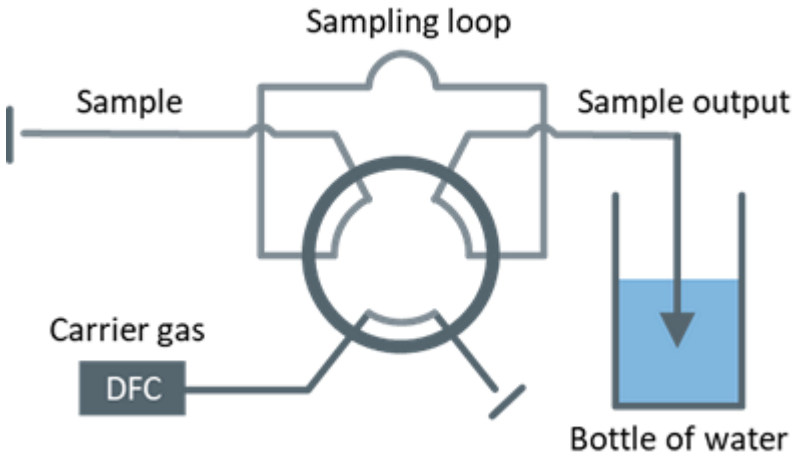


Figure 2.7 – “Overflow” check

2.7.4 Valve ports washing recommendations

Valve ports, tubing and loops washing should be carried out in following cases:

- Ports and lines are polluted with heavy components of analyzed sample;
- Analyzing sample liquid entered into valve ports;
- At ghost peaks of analytes (carryover), which are supposed to be in valve and connected gas lines.

Prior to ports washing it’s necessary to make sure that ghost peaks are results of device ports polluting, and not present in chromatograph ports or analyzing sample.

At washing the following recommendations should be considered.

Before washing, it is necessary to disconnect columns, traps and other elements of gas lines from the valve, solvent ingress into which can damage them.

- Only those gas line sections should be washed where sample components ingress is possible.
- Ready-mounted lines should not be washed. Prior to washing gas lines should be completely dismounted and wash each part (tube, valve port etc. separately).
- Solvent should be chosen properly guided by the following requirements. Solvent should dissolve supposed contaminants, with passive treatment of washing channels’ material, easily removable and be flushed by carrier gas. Preferable solvents are ethanol, acetone.
- After washing ports should be carefully flushed and dried by carrier gas.

3 DETECTORS

3.1 Thermal conductivity detector (TCD)

3.1.1 TCD operation principle

Principle of operation is based on registration of temperature changes between two heated filaments, which depends on thermal conductivity of gaseous flow, reflecting its composition. One of the filaments is flowed by pure carrier gas (reference gas), the second one is flowed by carrier gas with sample components coming from chromatographic column.

TCD has sensitivity to all compounds which thermal conductivity differs from thermal conductivity of the carrier gas. The greatest sensitivity can be received in case when thermal conductivity of analyzed substance strongly differs from thermal conductivity of the carrier gas. Helium, nitrogen or argon is usually applied as a carrier gas.

TCD is a non-destructive detector, so the sample passed through it can be further determined using other detectors.

The linear range of accidents is over 10^5.

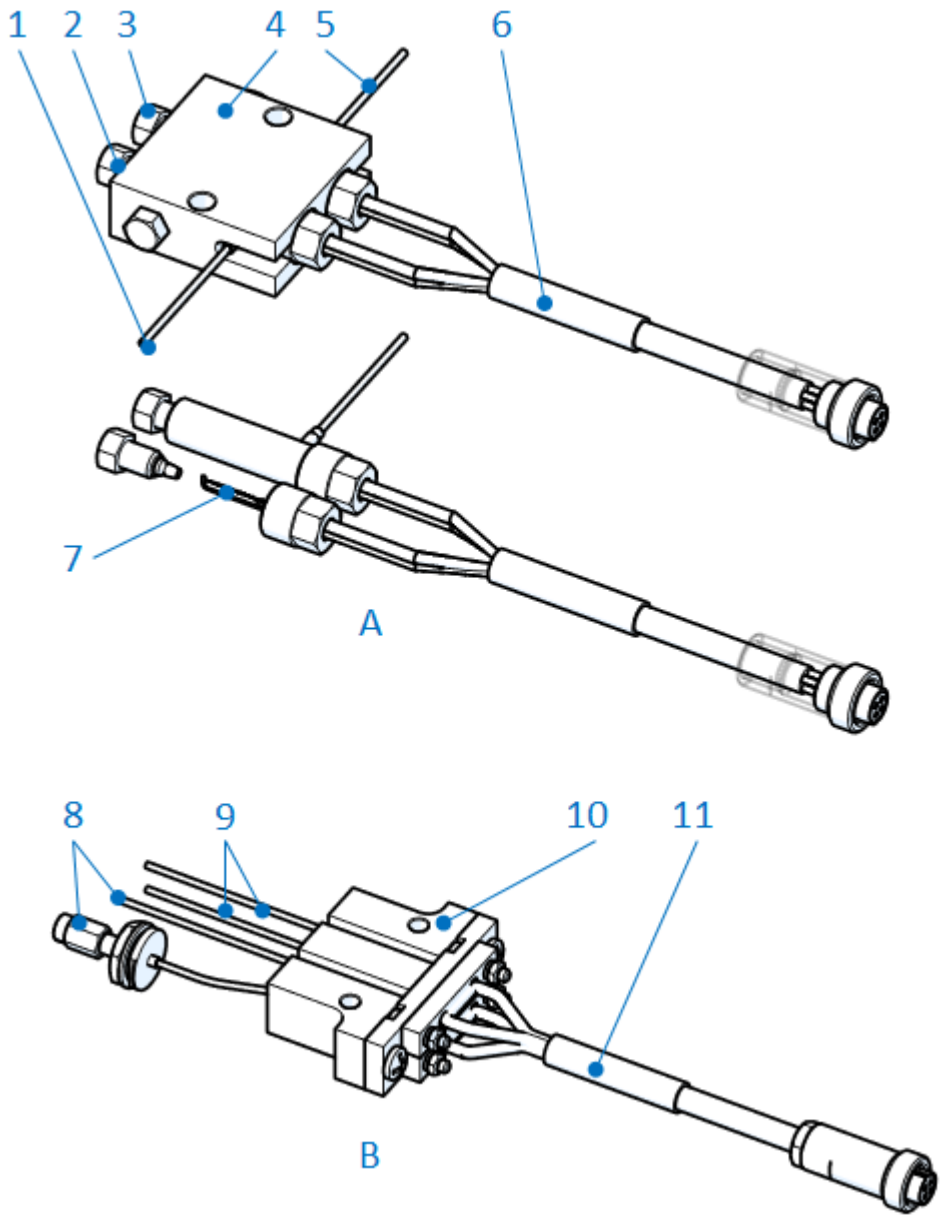
3.1.2 TCD construction

TCD represents is a metal block, in two chambers (cells) of which there are thermosensitive elements – a comparison element and a working element. A carrier gas with a sample is passed through the analytical cell, only the carrier gas is passed through the reference cell. The cells are equal in design, thus any of them may serve as an analytical cell, other will serve as a reference one.

Filaments are included in the measuring bridge, measured analogue signal is digitized and recorded in chromatogram.

There are several versions of TCD for various applications.

The design of all versions of TCD allows you to change the thermistors by the user at the workplace. The time of changing the thermistors of TCD t is no more than 5 minutes.



A – TCD 2.840.005-01.XX, B – TCD 2.840.005-05.00 (µ-TCD)

1,5 – TCD cells output, 2, 3 – TCD cells input, 4 – TCD body, 6 – TCD sensor, 7 – filament, 8 – µ-TCD cells input, 9 – µ-TCD cells output, 10 – µ-TCD body, 11 – µ-TCD sensor.

Figure 3.1 – Detectors TCD design

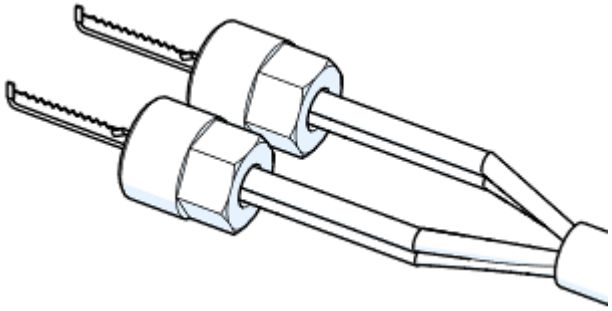
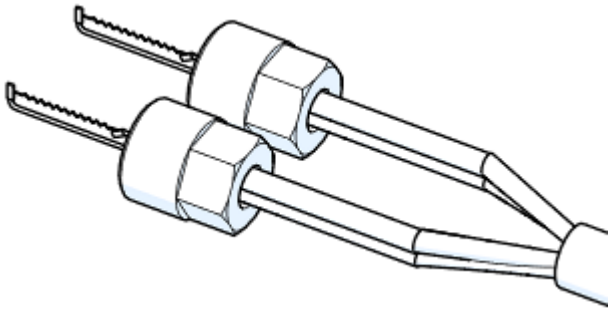
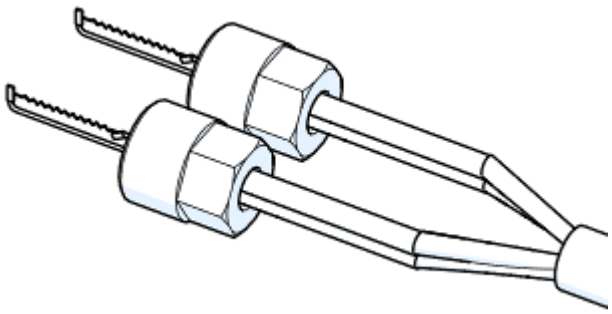
TCD detectors of P/N 150-2400 – 150-2425 have aluminum body; stainless steel cell and sensor may vary depending on modification. Detectors are recommended for operation with packed columns or wide-bore capillary columns (0.53 mm). Maximum operating temperature is 350 °C.

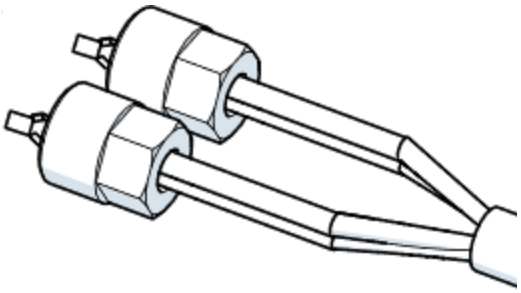
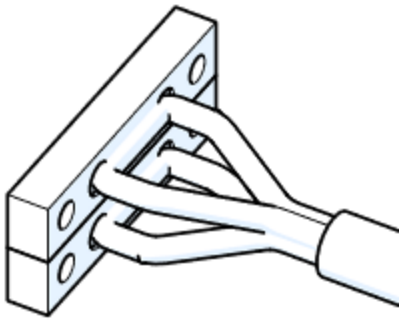
Detector TCD (P/N 150-2460) (micro-TCD) has aluminum body as well. Detectors cells made of stainless steel. The cell's operating volume is 10 µl. Detectors are recommended for operation with capillary columns (0.15 – 0.53 mm). Maximum operating temperature is 300 °C.

All TCD modifications have equal mounting dimensions.

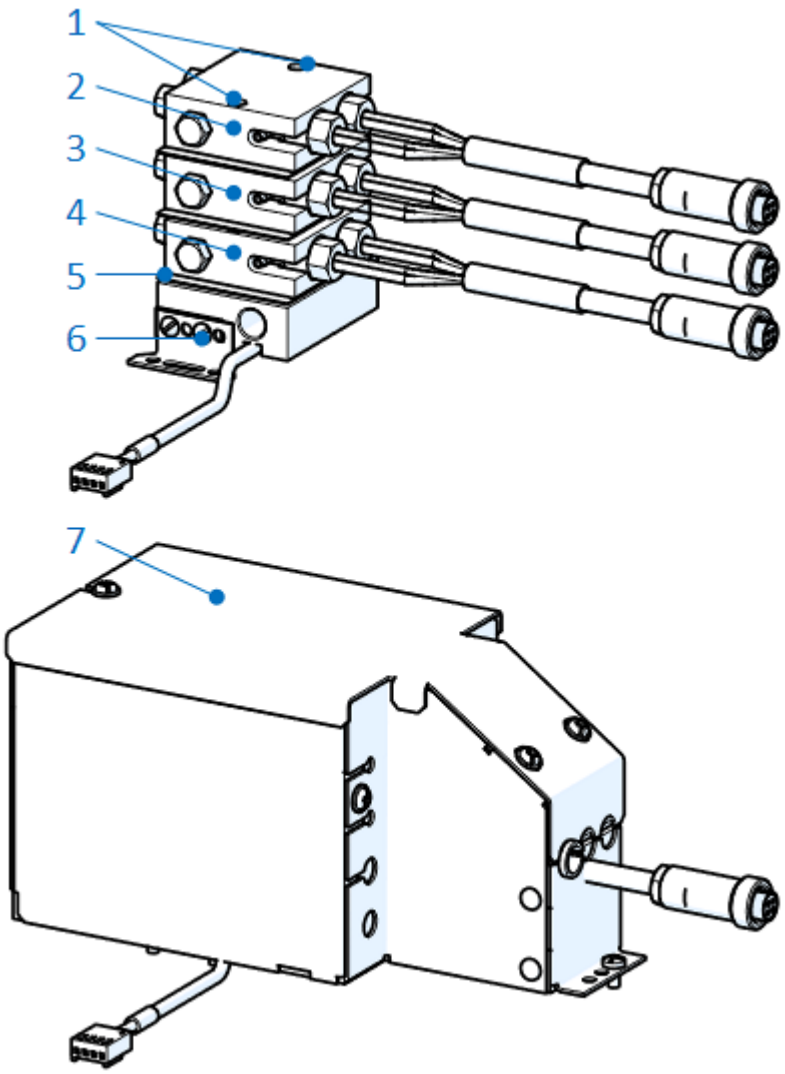
TCD body is placed into TCD house carefully temperature-controlled in order to obtain the best performance (flat baseline and best sensitivity). Power of TCD heater is 80 W. There are TCD modifications depending on sensors types and diameters of input/output tubes (Table 3.1).

Table 3.1 – TCD modifications

Part number	**Cell volume, mm3**	**Sensor type**	**Tubing (o.d. x i.d.), mm**	
			Input	Output
150-2400 150-2405	600	Sensor 5.863.149 Bi-spiral (W/Re) Tungsten-Rhenium alloy filaments Resistance is 60 Ohm at 20 °C  Sensor has average sensitivity and durability to oxidation	Ø1.6×1.0 Ø1.6×1.0	Ø1.6×1.0 Ø3.0×2.0
150-2410 150-2415	400	Sensor 5.863.149-02 Spiral Tungsten filaments Resistance is 19 Ohm at 20 °C  Sensor has the best sensitivity and average durability to oxidation	Ø1.6×1.0 Ø1.6×1.0	Ø1.6×1.0 Ø3.0×2.0
150-2411 150-2416	400	Sensor 5.863.149-03 Spiral Gold-coated Tungsten filaments Resistance is 24 Ohm at 25 °C 	Ø1.6×1.0 Ø1.6×1.0	Ø1.6×1.0 Ø3.0×2.0

Part number	Cell volume, mm3	Sensor type	Tubing (o.d. x i.d.), mm	
			Input	Output
		Sensor has the best sensitivity and durability to oxidation		
150-2420 150-2425	200	Sensor 5.863.149-01 Corrosion resistive sensor (CR) Resistance is 108 Ohm at 20 °C  Sensor has average sensitivity and excellent durability to oxidation. Sensor is not suitable for determination of hydrogen and helium using argon (nitrogen) as a carrier gas	Ø1.6×1.0 Ø1.6×1.0	Ø1.6×1.0 Ø3.0×2.0
150-2460	10	Sensor 5.863.226 Spiral Tungsten filaments Resistance is 40 Ohm at 20 °C  Sensor has average sensitivity and durability to oxidation	Ø1.6×0.25	Ø1.6×0.25

3.1.3 TCD montage



1 – fixing screws, 2 – TCD-3, 3 – TCD -2, 4 – TCD -1, 5 – mica-laminated plate, 6 – heating panel, 7 – cover.

Figure 3.2 – TCD detectors montage on the heating panel

Up to 3 TCD detectors may be installed on the single heating panel. There may be installed up to 2 heating panels for TCD in the GC.

3.1.4 TCD amplifiers

Types of sensors with significant difference in resistance, correspond to different amplifier types! Otherwise, sensor can be damaged.

Baseline shift value is applied for automatic bridge balancing. This (TCD shift) is recommended to set not less than 1000 mV.

Maximum baseline level at automatic bridge balancing is determined by particular pair of filaments in the sensor and usually exceeds 5000 mV.

Table 3.2 – TCD amplifiers modifications

Amplifier's type	**Sensors type, resistance**	**Marking**	**Input connector type**	**Dynamic range**	**Max. signal level, V**
5.173.039	5.863.149-01 R = 108 Ohm	TCD-QR-CR-1 TCD-QR-CR-2 TCD-QR-CR-3	PC-4 ATB or PC-4 TB	10^6 (quadratic amplifier)	100
5.173.032	5.863.149 R = 60 Ohm	TCD-QR-1 TCD-QR-2 TCD-QR-3	PC-4 ATB or PC-4 TB	10^6 (quadratic amplifier)	100
5.173.040	5.863.149-02 R = 18 Ohm 5.863.149-03 R = 24 Ohm 5.863.226 R = 40 Ohm	TCD-20-QR-1 TCD-20-QR-2 TCD-20-QR-3	PC-4 ATB or PC-4 TB	10^6 (quadratic amplifier)	100

3.1.5 TCD pneumatic connections

Typical feeding of TCD reference cell feeding is shown on figure below.

Reference gas flow rate is to be set equal to carrier gas flow rate passing through the analytical cell. To stabilize flow rate through the reference cell, Filter 5.884.065 is installed in front of the cell.

Reference cell is powered directly from the gas controller

Connection of flow controller DFC-10 to the reference cell.

When 2 detectors TCD operating with the same carrier gas are mounted in the GC, consecutive feeding may be applied.

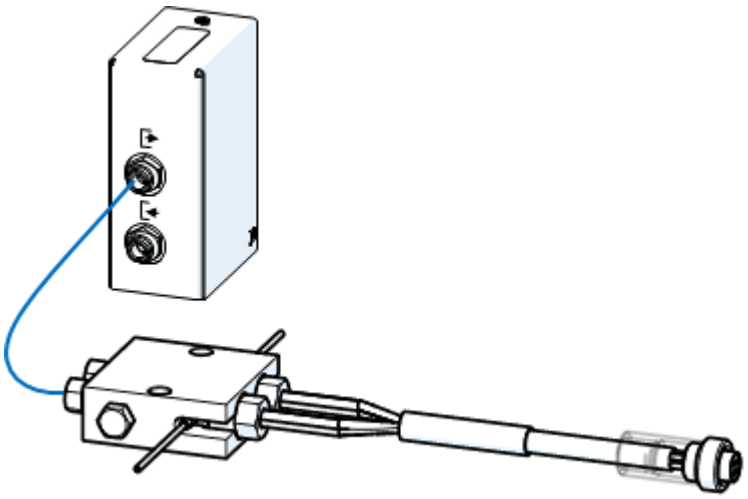


Figure 3.3 – TCD Flow diagram. Reference cell gas feeding

Analytical cell gas feeding

The inlet of the packed column can be connected to inlet or a sampling valve (Figure 3.4).

The flow of carrier gas through the capillary column for detectors 2.840.005-01.XX is usually insufficient to obtain acceptably narrow peaks. Therefore, the carrier gas is blown analytical cell from the flow controller DFC-10 (Figure 3.5). Blowing is carried out using the tee 6.453.131-03 in column oven (Figure 3.9).

When working with the detector 2.840.005-05.00 (micro-TCD), additional blowing of the carrier gas is not needed.

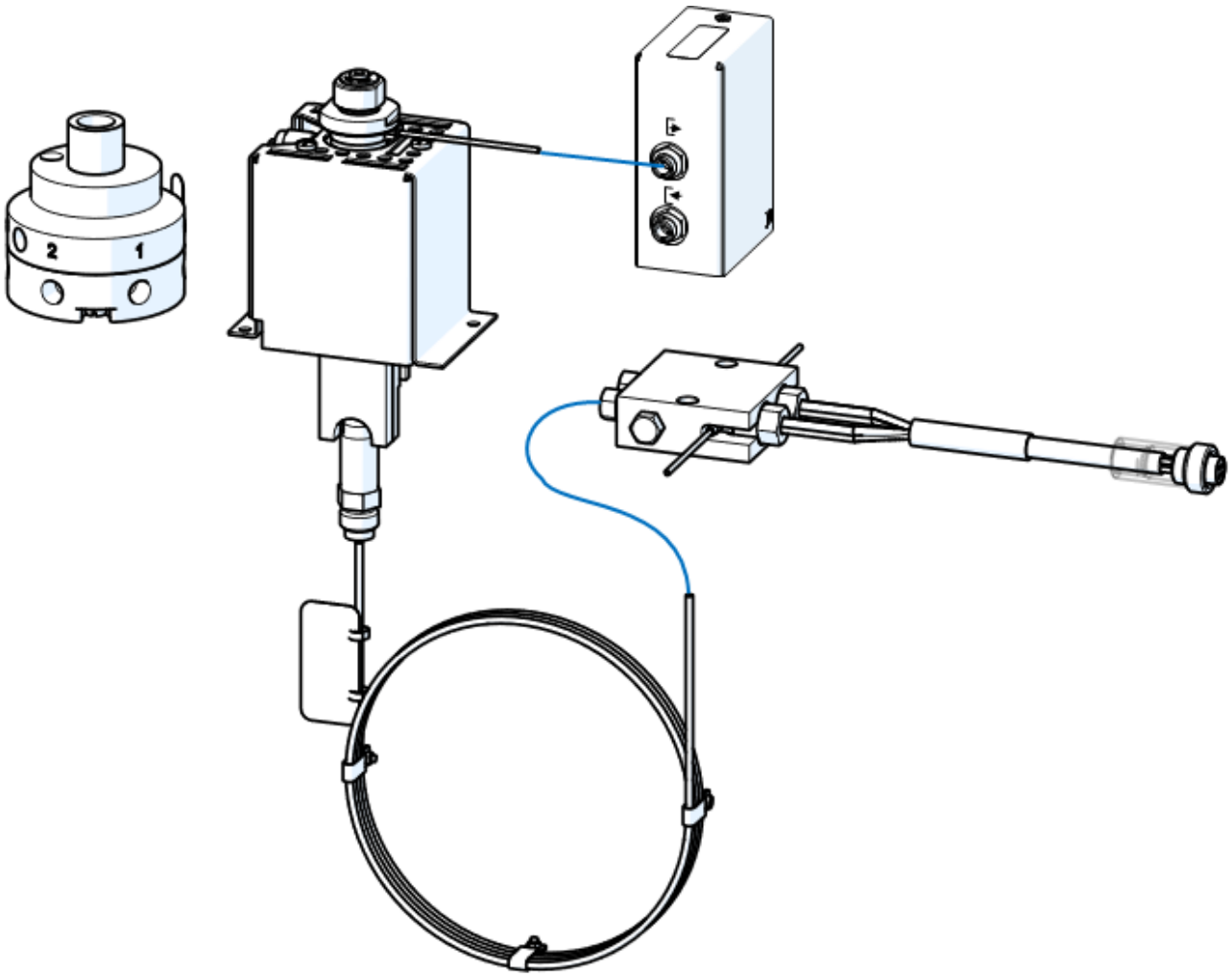


Figure 3.4 – TCD Flow diagram. analytical cell gas feeding

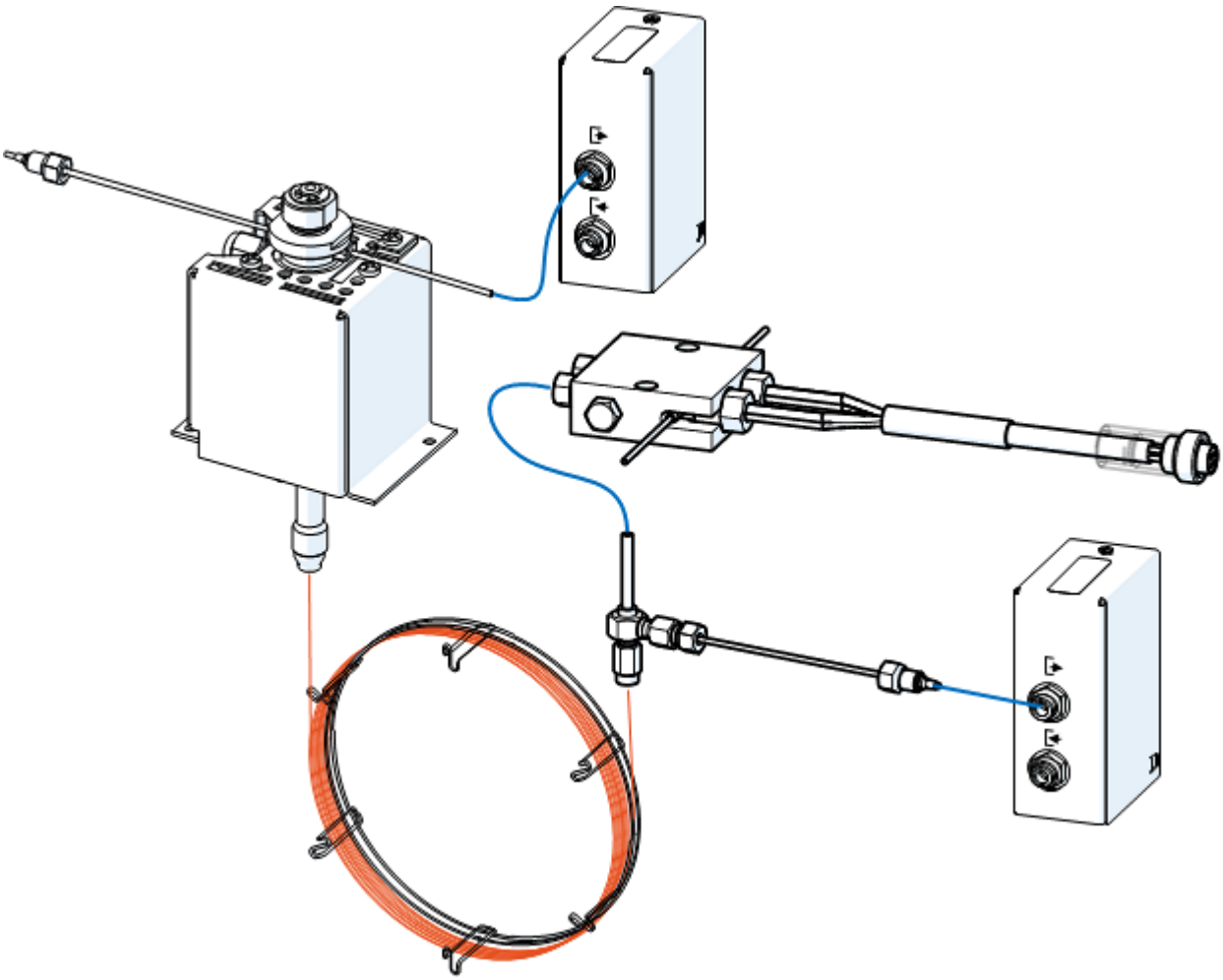


Figure 3.5 – Capillary column connection to analytical cell of TCD

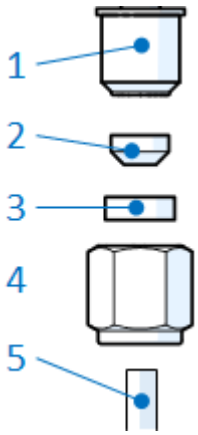
3.1.6 Columns connection to TCD

Packed column connection depending on column type and ferrules material is shown below.

Rubber ferrules are recommended for use at temperature in the oven less than 250 °C.

Assemble nut 8.930.172, insert 8.223.013 and rubber ferrule 8.220.380 onto the packed column end.

Insert the end of the column into valve's fitting up to the stop. Hold the column in this position and tighten the nut finger tight.



1 – Fitting, 2 – ferrule 8.220.380 (rubber), 3 – insert 8.223.013, 4 – nut 8.930.172, 5 – packed column

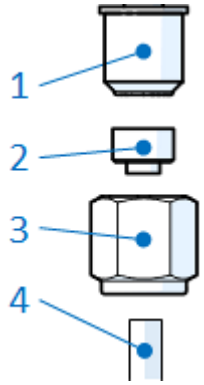
Figure 3.6 – Packed column connection using rubbery ferrule

Graphite ferrules are allowed to use in the oven up to 450°C.

Assemble nut 8.930.172, ferrule holder 6.453.038 onto the packed column end.

Insert the end of the column into detector's fitting up to the stop. Hold the column in this position and tighten the nut finger tight. Use a wrench to swage the nut at additional 30-60 degrees.

Be careful when tightening glass column with graphite ferrules in order to avoid its breakage.



1 – Fitting, 2 – ferrule 6.453.038 (Graphite), 3 – nut 8.930.172, 4 – packed column

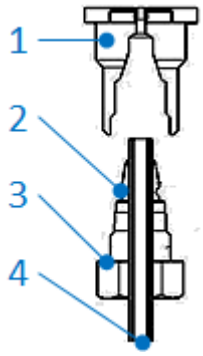
Figure 3.7 – Packed column connection using graphite ferrule

Type 3 columns with 3.0mm and 1/8’ connection diameter

To connect column of type 3 appropriate fitting should be installed in the oven.

To connect column of 3mm outer diameter ferrules 8.658.038-01 are to be used, for column of 3.18mm (1/8”) o.d. use ferrules 8.658.038-03.

Insert the end of the column into detector’s fitting up to the stop. Hold the column in this position and tighten the nut finger tight. Use a wrench to tighten the nut at additional 30-60 degrees.



1 – Fitting, 2 – ferrule, 3 – nut 8.930.114, 4 – packed column

Figure 3.8 – Packed column (type 3) connection using metal ferrule

Capillary column connection to TCD P/N 150-2400, #2410, #2420

To connect the capillary column to the detectors 2.840.005-01.XX, T-branch fitting 6.453.131-03 (item 4, Figure 3.9) or T-branch fitting 6.453.131-01 (to fitting for packed column type 3 (3 or 3.18 mm)) is used.

Nut 3 (8.930.172) and ferrule 2 (6.453.038) are installed sequentially on T-branch fitting 4, as shown in the figure. T-branch fitting is inserted into TCD 1 fitting until it stops, and is sealed with a nut 3. The tightening force of the nut is "by hand", followed by a 180–270-degree rotation for a new ferrule and 45-60 degrees for repeated tightening.

Side tube 5 with a diameter of 1.6 mm with ferrule 8.658.039-01 and nut 8.930.161 is connected to the carrier gas TCD blowing line.

Column connection

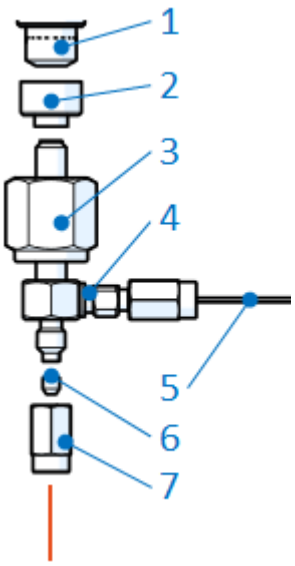
Assemble nut 7 (8.930.339) and ferule 6 (for example, #072626 SGE, cone down) on the end of the column.

After that cut 5-20 mm length from the end of column. The cut should be flat without chips and scratches. Wipe cut end of the column with lint-free issue wetted in alcohol in order to remove dust and other contaminants.

Put nut 7 and ferrule at a distance 30 – 40 mm from the bottom edge of the nut to the end of the column (above the level of the side fitting). For convenience, the distance could be fixed by marker slightly below nut, or with the help of rubber septum.

Insert the column into T-branch fitting. Tighten nut 7 by hand, then adjust column position precisely by marking or septa. Tighten the nut by wrench at 60-90 degrees so that column cannot be withdrawn from the fitting by fingers (column is tightened by ferrule).

The tubing for “make up” gas is connected in the lateral fitting of T-branch fitting in the same way.



1 – Fitting, 2 – ferrule 6.453.038, 3 – nut 8.930.172, 4 – T-branch fitting 6.453.131-03, 5 – “make up” gas tubing, 6 – ferrule, 7 – nut 8.930.339

Figure 3.9 – Capillary column connection

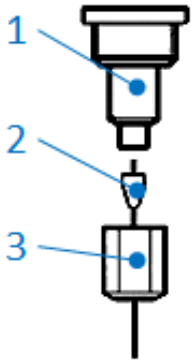
Capillary column connection to micro-TCD 150-2460

Capillary column is connected to detectors fitting directly. Depending on column diameter Ferrules Vespel-Graphite P/N 072663 (SGE) (hole 0.4 mm, for 0.25 mm i.d.), P/N 072654 (hole 0.5 mm, for 0.32 mm i.d.), or P/N 072655 (hole 0.8 mm, for 0.53 mm i.d.) are to be used for tightening.

Fit nut 8.930.339 with reversed cone onto the column end, then fit ferrule with its cone downward.

Column shall to be inserted up to the stop (0.8mm from the ferrule edge to column end).

Tighten nut 8.930.339 by hand, then tighten with wrench at 45-90 degrees (depending on column diameter), so that column cannot be withdrawn from the fitting by fingers.



1 – TCD fitting, 2 – ferrule, 3 – nut 8.930.339

Figure 3.10 – Capillary column connection to micro-TCD

3.1.7 TCD application features

TCD modifications with input and output tubes of 1.6 mm o.d. – 1 mm i.d. (P/N 150-2400, #2410, #2411, #2420) are recommended for analyses of gaseous samples and any volatile compounds (most applications).

TCD modifications with output tubes of 3 mm o.d. – 2 mm i.d. (P/N 150-2405, #2415, #2416, #2421) are recommended for analyses of high boiling compounds as well as for low boiling point liquid phase applications.

Micro-TCD (P/N 150-2460) is applied for operation with capillary columns in flow range from 1 to 8 ml/min.

When using programming temperature in the oven and packed columns DFC-10 are recommended to control carrier gas flow. It guarantees constant mass flow of the carrier gas through the column while increasing restriction of the column. Otherwise, when light components with retention time close to dead time analyzed, DPC-11 in the “pressure” mode is recommended. In this case pressure level is adjusted to obtain required flow rate the same as through the reference cell. Using pressure regulator allows to even background signal dip caused by sample injection.

Thermal Conductivity Detector is the concentration detector. The response of detector to component depends on its concentration in the cell. Therefore, the more carrier gas flow rate through detector analytical cell, the lower analyte concentration in this flow. So minimal value of “make up” gas is recommended to gain better sensitivity. Do not confuse two opposite ratios: response dependence on flow through detector (inverse proportionality) and component height dependence on the column flow (direct proportionality).

Figure 3.11 shows approximate dependence of peak height on carrier gas flow through detector analytical cell for column with 2 mm inside diameter for light hydrocarbons.

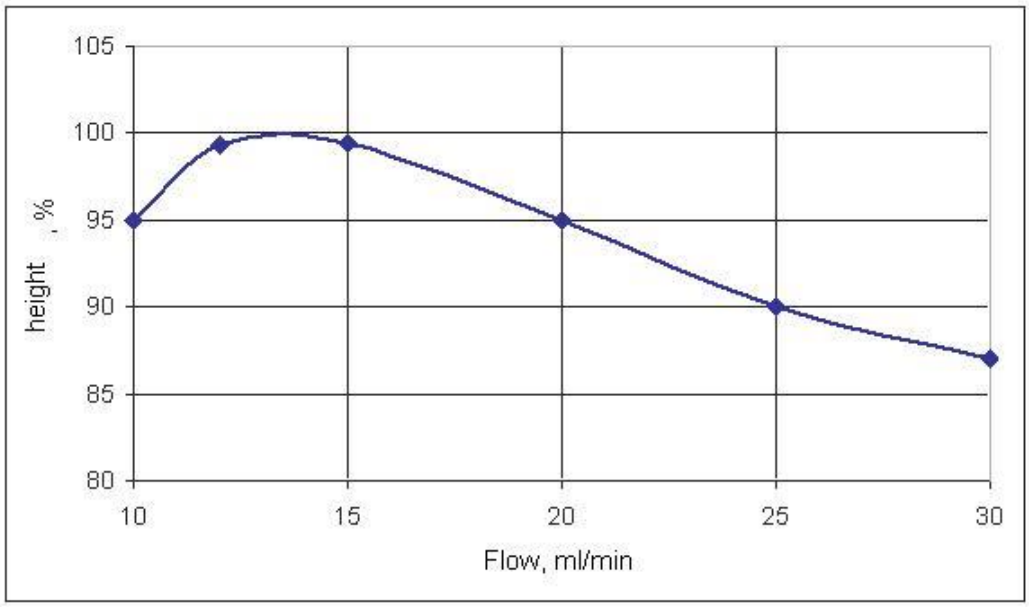


Figure 3.11 – Dependence of peak height on carrier gas flow rate

The best sensitivity of TCD can be achieved if difference in thermal conductivity between carrier gas and analyzed component is as much as possible. These gases are hydrogen (for most components) and argon (for determination of hydrogen, helium, neon). Helium is widely spread as a carrier gas instead of hydrogen hence its better operational safety.

There are some specific components pair, displaying non-typical peaks on TCD. At analysis of hydrogen using helium carrier gas, hydrogen peak may appear as:

- Negative at hydrogen high concentration;
- Positive at hydrogen small concentration;
- Split shape of peak at average hydrogen concentration.

At positive peaks for hydrogen using helium carrier gas the relative factor of detector sensitivity can differ on the order from factor of other components sensitivity. Argon or nitrogen is recommended as a carrier gas for hydrogen determination.

3.1.8 Protection of TCD filaments from oxidation

To protect the TCD filaments from irreversible oxidation chromatograph is provided with the following features.

- Protection of TCD filaments from overheat when operated in full power (100 %). If thermal conductivity of carrier-gas is different from the nominal value due to the change in its composition or if there is no flow the filaments switch off. This protection is inactive for CR sensors 5.863.149-01 (108 Ohm at 20°C) when the selected power is 25% and at the "analysis" stage.

- Filaments switch on automatically only when all gas flow rates reach the specified mode, TCD temperature reaches the preset value and at least 15 minutes after turning on the device and setting GC mode.
- When the flow rate of any gas selected in the analysis method is less than 5 ml/min for more than 1 minute the message "out of range" displays and at the same time filaments switch off. After carrier gas flow is recovered filaments switch on automatically. This happens at any operating state or mode.

3.1.9 TCD maintenance

If TCD specifications comply with the requirements of chromatograph test method then TCD operates correctly. Therefore, if you hesitate the detector operates correctly first compare the respective indicators with the rated values from the test method and only after that make the decision.

One of the signs of correct detector operation is an even noise structure with no spikes, jumps and irregular drift as shown on the picture.



Figure 3.1 – Typical TCD noise

This section describes the possible failures, remedy actions and preventive measures.

Check for resistance of filaments

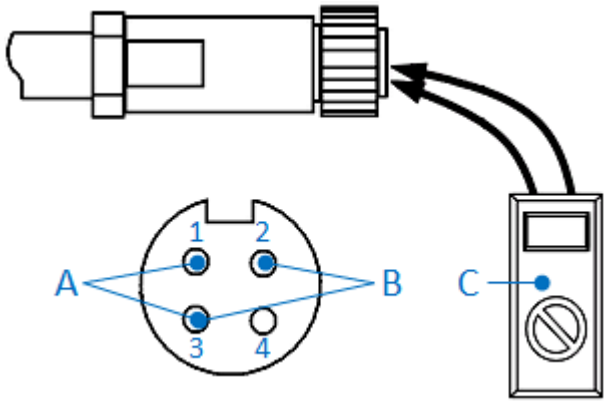
When manufactured filaments are joined into pairs according to their rated values with resistance difference up to 0.10 Ohm. When the detector is operated the resistance difference (unbalance) will increase with the time. Therefore, it should be checked on the regular basis. It is recommended to change the elements when the resistance difference is max 0.3 Ohm. However, the detector can operate when the unbalance reaches 0.5–0.8 Ohm. If the amplifier cannot reach the recommended balance of 1000 mV the unbalance is critical. In such a case filament should be definitely replaced.

The **resistance check of** filaments should be carried out only when the detector temperature is stabilized. To perform the check first de-energize the device and then wait for it to reach the room temperature. To perform the resistance, check warm up the chromatograph at least for 30 min to reach the operating temperatures and follow all the safety precautions.

Check for resistance of TCD filaments

- If the check is to be carried out while the detector is heated, turn off TCD filaments in software;
- Remove right side cover of the GC;
- Remove cable from TCD amplifier;
- Measure resistance of filaments as shown in figure below;

If the difference of filaments resistance exceeds 0.5 Ohm, it is recommended to replace filaments



A – element 1, B – element 2, C – multimeter

Figure 3.2 Check for resistance of TCD filaments

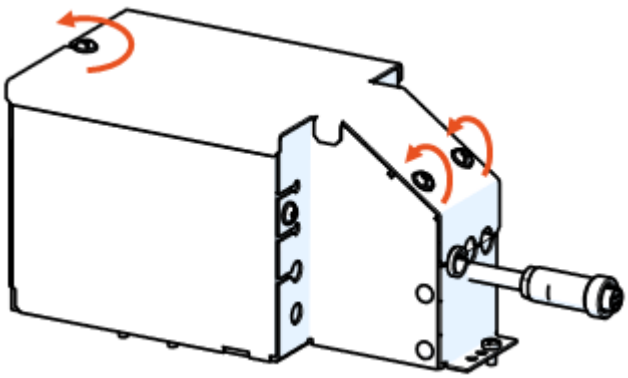
Filament replacement

Before replacement of the filament, it's necessary to turn the GC off and wait until the detector is cooled down to avoid burns.

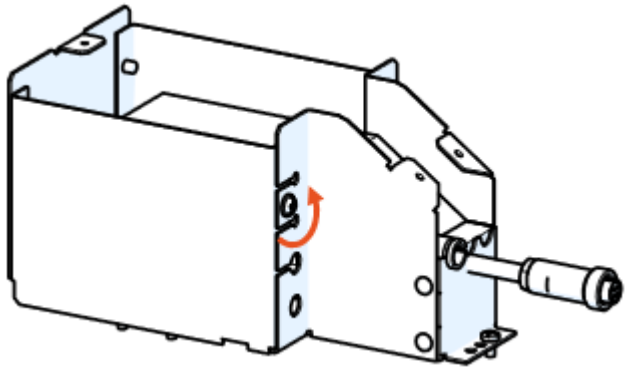
Keep record all procedures while disassembling, this allows to make easier the following assembling.

Replacement of filament in TCD detectors P/N 150-2400…2425:

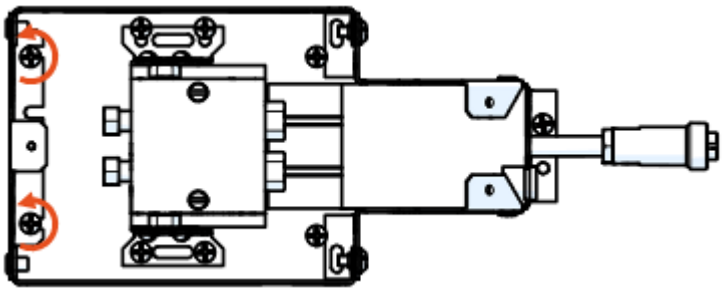
- Remove all protecting covers and thermal insulation; at this remember insulation folding order;
- Weaken left screw and unscrew both right screws. Then shift the top cover to the right slightly and remove it.



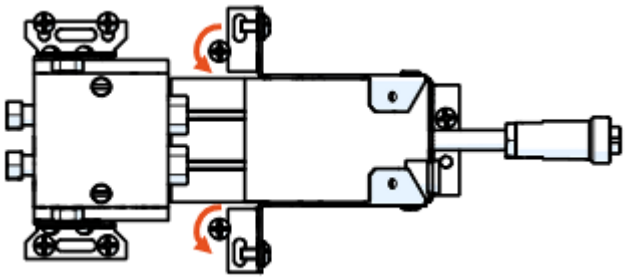
- Remove thermal insulation and unscrew two screw on the right side of cover.



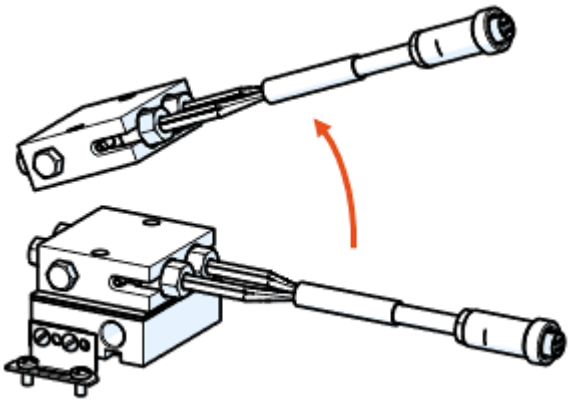
- Weaken two screws inside casing tightening left cover, then remove it.



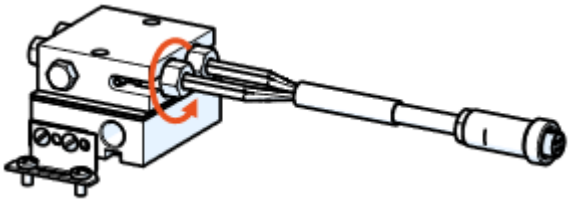
- Weaken two screws inside casing tightening right cover, then remove it.



- Remove right cover of GC, then disconnect cable from TCD amplifier.
- If there are 2 or 3 detectors on heating platform and one need to replace filaments in bottom detector, do unscrew two screws fixing all the detectors to heating platform and bow to the left upper detectors.



- Unscrew two nuts sealing TCD filaments in cells and eliminate them carefully. Previously lube the threaded connections with alcohol, and remove the elements. If the nuts could not be unscrewed, then the detector must be replaced entirely;



- After filament disconnection, blow detectors cells carefully and check visually the absence of strange particles;
- Insert new filament, taking into account parallelism of filaments location in cells.
- Nut, sealing filaments, should be screwed by fingers up to stop, then tightened at 45˚ with wrench. If leaks remained, tighten at 10-20o more and check for leaks again. Tightening nuts is recommended to do on removed detector stamped in vice or adjustable wrench.
- Assemble the detector in reverse order.

Check for leaks

The leak in the TCD flow paths reduces the life cycle of filaments and increases the analysis error rate.

The check for leaks is carried out every time after assembly/disassembly of any flow diagram element, after the device transportation, when putting into operation after long downtime and also when detecting any indirect signs of a possible leak in the course of analysis.

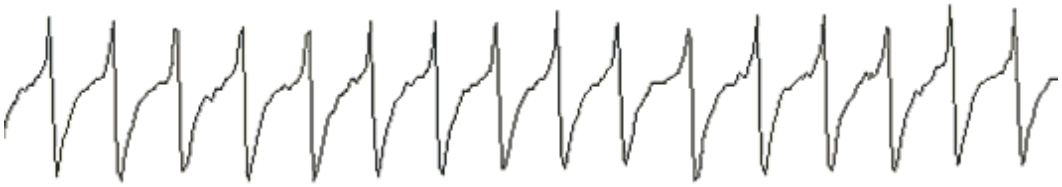
Procedure:

- Select **Test** mode in the device configuration and switch off the detector filaments to check the TCD flow paths for leaks.
- Referring to the flow diagram insert the plugs from the device spares kit into every possible carrier-gas outlet. It is acceptable to check separate flow paths for leaks rather than the whole flow diagram at once. Normally, the path used to deliver the sample into chromatograph is checked individually.
- Set the carrier-gas pressure at the inlet 0.1 MPa higher than the one normally set for operation.
- For everyDFC-10 set the carrier-gas flow rate at 100 ml/min, for every DPC-11 – 400 kPa and wait until the measured flow rate values stabilize in the respective DFPC channels. The flow should sink to 0–0.1 ml/min providing that the DFPC itself is leak tight. In case of DPC-11 the leak tightness can be checked by the pressure drop in the line. The check by pressure drop is only possible if the temperature of ambient air (no drafts or air conditioning) and tested paths are stabilized. The tested flow path should be connected to DPC-11 outlet. Set the regulator pressure to 400 kPa. Wait until the flow rate stabilizes at about 0 ml/min. Then set the DPC-11 pressure to 0 kPa and monitor the pressure drop in the line. The acceptable pressure drop is max 2 % (8 kPa) from the original value within 30 minutes.

The possible leak points (normally, connections and welded seams) should be checked by gas leak-tester, dripping ethanol, acoustic leak-tester etc. It is not recommended to use the foaming solutions in order to prevent the foam from penetrating tubes and affecting the device operation. However, such solutions are still acceptable in urgent cases. Carefully wipe the foam after use.

Detector washing

In some cases when operating TCD, liquid condensate can form inside the detector exhaust piping resulting in spikes (up and down) that prevent the detector from correct operation.



Condensate forms in the following cases:

- when performing the analysis using packed columns filled with sorbent with high concentration of liquid phase at temperatures close to maximum acceptable for this sorbent,
- when samples containing a large number of components analyzed, with boiling temperatures above 100–150 °C. Due to condensate in tubes.

To prevent the formation of condensate it is recommended to use TCD modification with wide heated outlet TCD pipes slightly sloped down to the horizontal line.

To remove the condensate washing the detector is required. The detector design allows washing both by polar (e.g., acetone) and non-polar organic solvents (e.g., hexane).

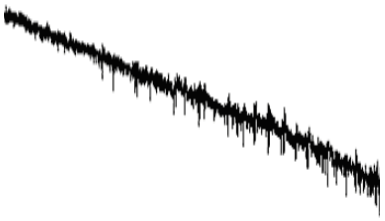
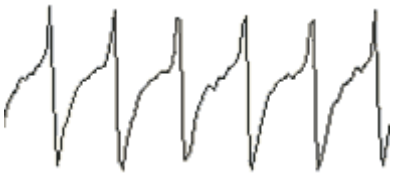
Procedure:

- Disconnect GC plug from the power supply, Temperature zones of the detector and other areas must be cooled down to ambient.
- disconnect the outlet of analytical column from the detector inlet,
- prepare a syringe (up to 20 ml volume) with a needle, any type of rubber septa to provide the leaktight joint with detector inlet connector (e.g., inlet septum), fill in the syringe with acetone and securely connect it to the detector, wash the detector with solvent.
- collect the leaking solvent at the detector outlet;
- right after the washing blow the detector with carrier-gas or air to remove the solvent residues,
- connect analytical column,
- connect GC plug to power supply, switch it on, set the operating mode, wait for the solvent residues to blow out.

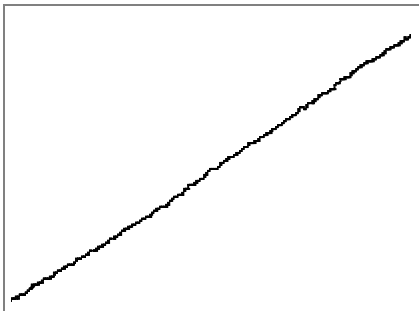
Avoid exposing the detector to solvent for a long time.

Avoid washing the detector as described above if the solvent may ingress the valves or other units containing fluoroplastic elements (refer to flow diagram if required).

Troubleshooting

Failure	**Possible cause**	**Remedy action**
Increased noise level 	Electronic noise	Connect special equivalent to the amplifier. Replace the TCD amplifier and/or GC power adapter after confirming the electronic origin of the noise.
	Thermal instability	Check the heat insulation around the detector to make sure there are no cracks. If necessary, add more insulation. And/or replace the filament. And/or make sure GC power supply is stable.
	Unstable gas flow	Replace the pressure regulator on the carrier-gas cylinder. And/or replace the DFPC. And/or replace/clean the filters. Check the leak tightness. Restore it, if necessary.
	Bleeding from the column or tube	Replace the carrier-gas. Condition or replace the column. Replace the tube.
	Vibrations	Eliminate the vibrations
	Unstable operation of the filaments	Replace the filaments
	Influence of adjacent devices connected to the same gas line	Connect the devices to the separate gas lines. And/or install the additional pressure regulators at the inlet of feeding gases into the device.
Regular spikes up and down on chromatogram 	Condensate inside the outlet tube	Wash the detector
	Sudden indoor pressure change	Install special pneumatic resistances with buffer tank at the TCD outlet.
	Unstable gas flow Influence of adjacent devices connected to the same gas line	Actions described above.
TCD filament does not switch on	The respective conditions are not met: gases or TCD	Increase the carrier-gas pressure at the device inlet, wait until gases and

	temperature is out of range, less than 15 minutes passed after turning on the GC and setting the mode	temperatures reach the specified parameters, wait for 15 minutes
	Filament overheat	Check the leak tightness. Specify the filament power according to the carrier-gas used. Adjust the carrier-gas flows through the working and reference cells. Wait until the air evacuates from the carrier-gas line and then switch on the filaments.
Amplifier cannot reach the balance	Filaments unbalanced	Check resistance of the filaments. Replace the filaments, if necessary.
	Different carrier-gas flows through the working and reference cells	Adjust the carrier-gas flows
Poor sensitivity	Filaments are switched off	Switch on the filaments
	Filament's power does not match with the carrier-gas used	Set the power to 100 % when using helium or hydrogen as a carrier-gas
	Detector temperature is too high	
	Leak	Spot the leak and eliminate it
	The volume of injected sample is lower than required	Increase the volume
	Carrier-gas flow is too high	Optimize the operating mode: reduce the flow, increase the column temperature
Monotonic drift 	TCD temperature changes	Wait until the detector temperature stabilizes
	Carrier-gas purity change	Replace/clean the filters. Replace the cylinder
	“Heavy” components come out of the column	Condition the column
	Leak	Spot the leak and eliminate it

	Change of carrier-gas flow through the cells	Adjust and stabilize the flows through the working and reference cells
	Change of the carrier-gas filter temperature.	Stabilize the filter temperature.
No repeatability between repeat runs	Leak	Spot the leak and eliminate it
	Unstable sample	Ensure the stable one-phase sample flow through sampling device.
	Sample sorption in gas lines	Replace the gas lines with sample-inert ones.

To perform some operations, such as replacement of filaments, it is recommended to contact the manufacturer or authorized representative.

Contact the manufacturer to get the advisory support and find solutions to other issues.

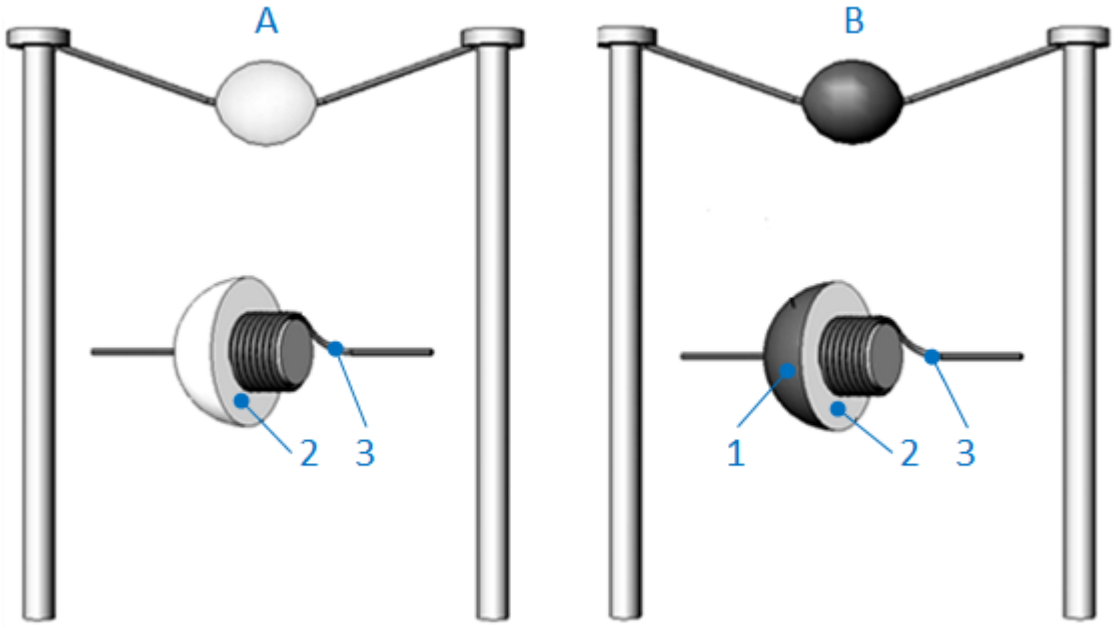
3.2 Catalytic combustion detector (CCD)

3.2.1 CCD operation principle

Detector is intended for determination of flammable gases and oxygen. Particularly, it can be used for determination of hydrogen in helium and oxygen in argon.

Operating principle is based on measuring of thermal effect at catalytic combustion of sample on the platinum sensors.

CCD contains catalytic and force-balance filaments connected to Wheatstone bridge. The filament is a spiral platinum fiber embedded into ceramic bead 1mm in diameter. Force-balance filament's bead doesn't have any coat (white color). Catalytic filament's bead is coated with Pt–Pd–catalyst having operating reaction temperature (400–500) ˚C (black color).



A – Force balance filament, B – Catalytic filament

1 – Catalytic coat, 2 – Ceramic bead, 3 – Platinum bead

Figure 3.12 – CCD filaments

While sample passes through the cell, its molecules impact the hot bead and combust on the surface raising the temperature and resistance of the platinum fiber. The resistance change causes the detector output signal to change thus producing a peak. Resistance variation is proportional to analyte concentration.

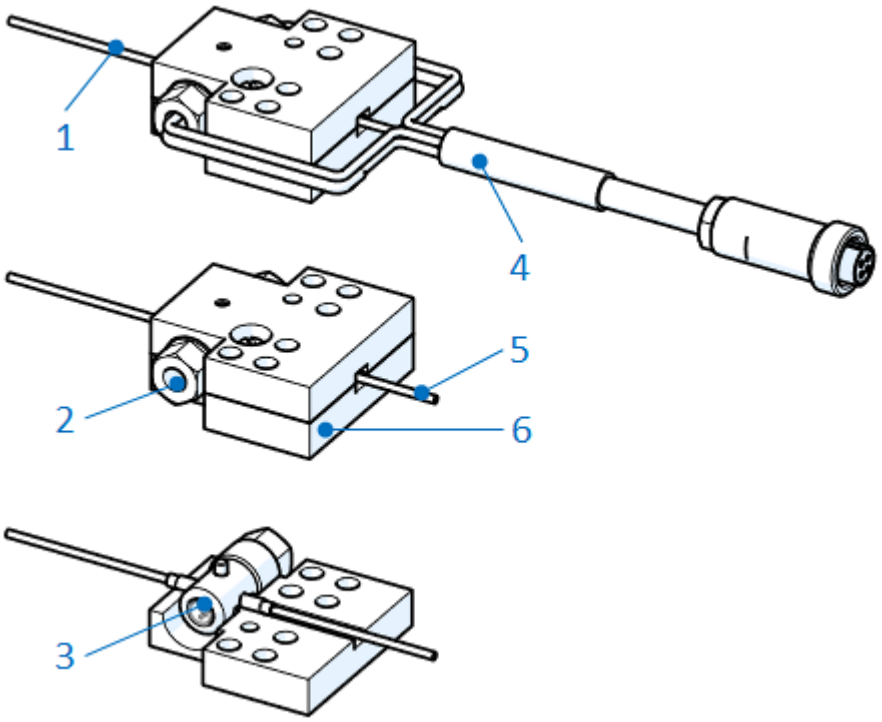
To start combustion on the catalyst surface RedOx reaction should be created. Therefore, oxygen is necessary to add to analyze flammable gases (hydrogen, hydrocarbons) and hydrogen should be blown into stream to determine oxygen.

For standard substances and test conditions, the linear range of CCD is over 10^3.

CCD 214.5.184.023-01.00 construction

Figure 3.13 shows CCD design.

Catalytic and force-balance filaments are mounted in Sensor 214.5.863.173, which is located in cell 214.5.887.032 with input and output tubes. The cell 214.5.887.032 is placed into aluminum casing consisting of two halves fixed with screws.



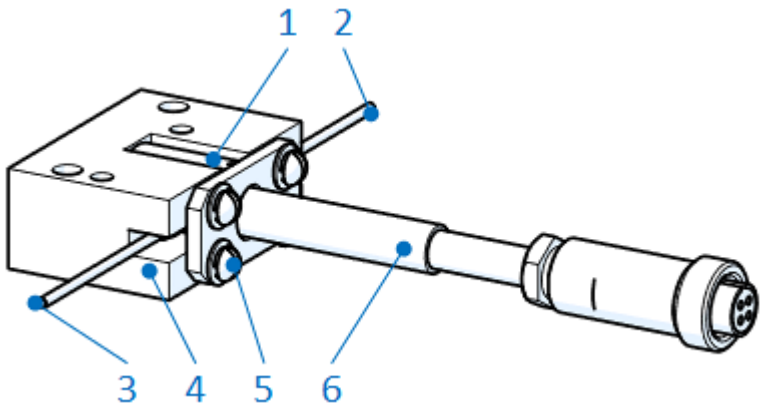
1 – Input, 2 – nut 8.930.312, 3 – CCD cell 5.887.032, 4 – Sensor 5.863.173, 5 – Output, 6 – CCD body

Figure 3.13 – CCD (P/N 150-2701) construction in section

There could be up to three detectors CCD located on one heater. CCD detectors can be installed on same heating panel together with TCD detectors.

CCD 214.5.184.023-02.00 construction

Catalytic and force-balance filaments are mounted in Sensor 214.5.863.205, which is located in detectors cell and fixed with 4 screws (Figure 3.14). Rubber O-ring is used as a sealing element. **CCD 214.5.184.023-02.00 maximum temperature is 80 °C.**



1 – CCD cell 5.887.046, 2 – output, 3 – input, 4 – CCD body 8.034.921, 5 – Sensor fixing screws, 6 – CCD sensor 5.863.205

Figure 3.15 – CCD (P/N 150-2711) construction

3.2.2 CCD pneumatic connections

Possible pneumatic diagrams of connection are shown below.

Sample is injected with packed inlet or sampling valve. Column's outlet is connected CCD fitting in the oven. T-branch 6.617.014-01 is installed on sample path way to add reagent gas.

At consecutive order TCD-CCD, T-branch is located in the same way after TCD.

If reagent gas is used as a carrier gas, T-branch is not used.

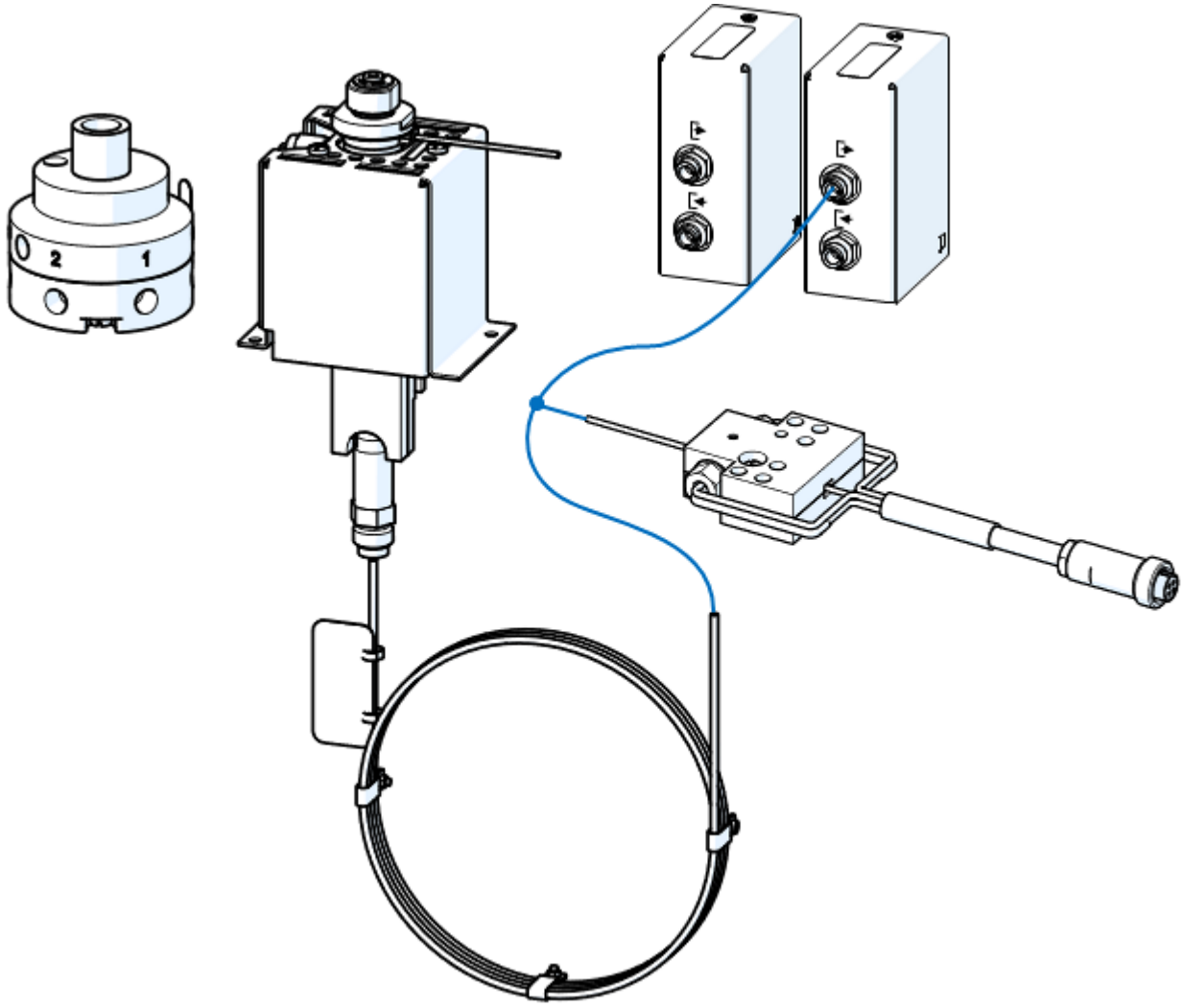


Figure 3.16 – Packed column connection to CCD

3.2.3 Packed column connections to CCD

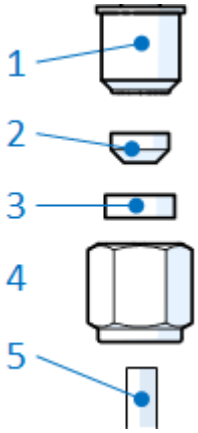
Packed column connection depending on column type and ferrules material is shown below.

Columns with 5.5 mm end diameter (type 1)

Rubber ferrules are recommended for use at temperature in the oven less than 250°C.

Put nut 8.930.172, insert 8.223.013 and rubber ferrule 8.220.380 on the packed column end.

Insert the end of the column into CCD fitting up to the stop. Hold the column in this position and tighten the nut finger tight.



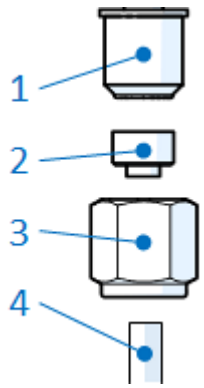
1 – Fitting, 2 – ferrule 8.220.380 (rubber), 3 – insert 8.223.013, 4 – nut 8.930.172, 5 – packed column

Figure 3.17 – Packed column connection using rubbery ferrule

Graphite ferrules are allowed to use in the oven up to 450°C.

Insert the end of the column into detector's fitting up to the stop. Hold the column in this position and tighten the nut 8.930.172 finger tight. Use a wrench to tighten the nut at additional 30-60 degrees.

Be careful when tightening glass column with graphite ferrules in order to avoid its breakage.



1 – Fitting, 2 – ferrule 6.453.038 (with graphite), 3 – nut 8.930.172, 4 – packed column

Figure 3.18 – Packed column connection using graphite ferrule

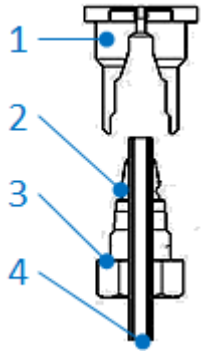
Connection of the packed column in column oven depending on the type of column and ferrules material is shown below.

Columns with 3.0 mm and 1/8'' end diameter (type 3)

To connect column of type 3 appropriate fitting should be installed in the oven.

To connect column of 3mm outer diameter ferrules 2 (8.658.038-01) are to be used, for column of 3.18mm (1/8") o.d. use ferrules 8.658.038-03.

Insert the end of the column into detector's fitting up to the stop. Hold the column in this position and tighten the nut finger 3 tight. Use a wrench to tighten the nut at additional 30-60 degrees.



1 – Fitting, 2 – ferrule, 3 – nut 8.930.114, 4 – column

Figure 3.20 – Packed column (type 3) connection using metal ferrule

3.2.4 CCD application features

CCD temperature is usually set according to test method requirements. Keep temperature level at least at (50–60) °C for better stabilization. Temperature level doesn't have any influence on sensitivity, particularly hydrogen response changes less than 2% within temperature range from 60 to 160°C.

CCD 214.5.184.023-02.00 maximum temperature is 80 °C

When helium and hydrogen are used as carrier gas, CCD 214.5.184.023-02.00 has very high noise and it is not recommended to work on them.

CCD detector 214.5.184.023-01.00 does not detect methane, CCD 214.5.184.023-02.00 detects methane.

Dependence of hydrogen response on reagent gas flow level (air) is shown below (Figure 3.). Maximum response is obtained at air flow rate (10–20) ml/min. At air increasing both area and height go down equally.

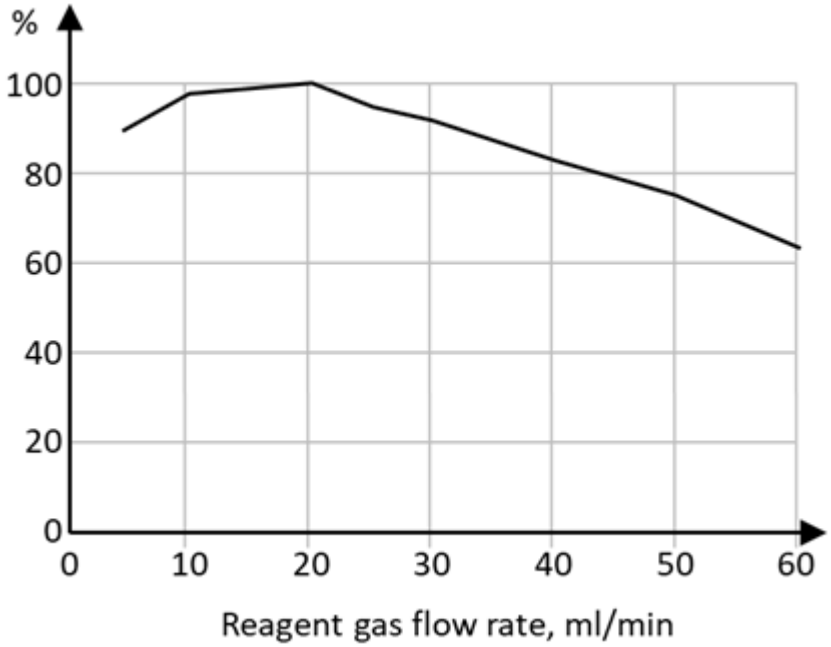


Figure 3.21 – Hydrogen response dependence on air flow rate

A negative fluctuation on the rear peak side may appear at air flow less 20 ml/min.

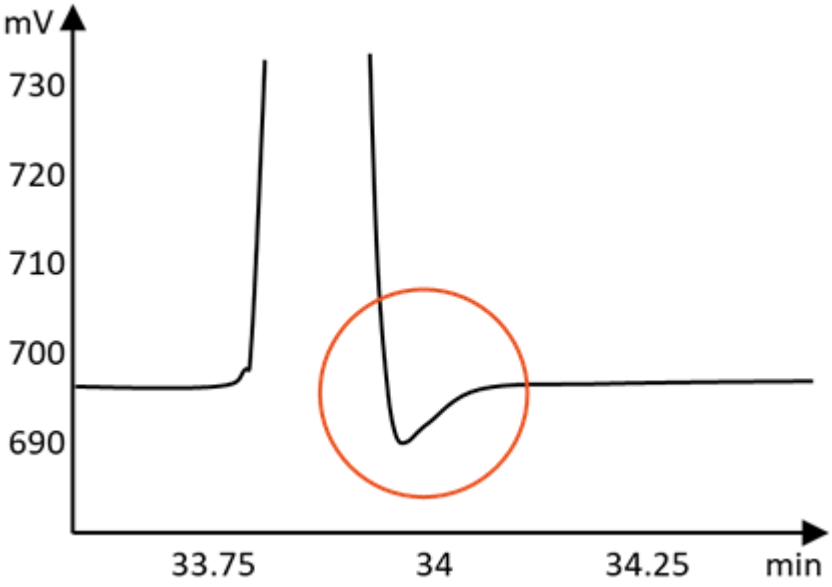


Figure 3.19 – Hydrogen peak's distortion at low air flow rate

Distortion disappears at higher flow rate 25 – 30 ml/min. Therefore, at hydrogen analysis, the optimal air flow rate is 30 ml/min.

3.2.5 CCD maintenance

All maintenance work on the detector should be carried out with the chromatograph turned off.

CCD detector can be washed only after sensors have been removed!

Washing CCD with sensors damages the sensors.

Check for resistance of filaments

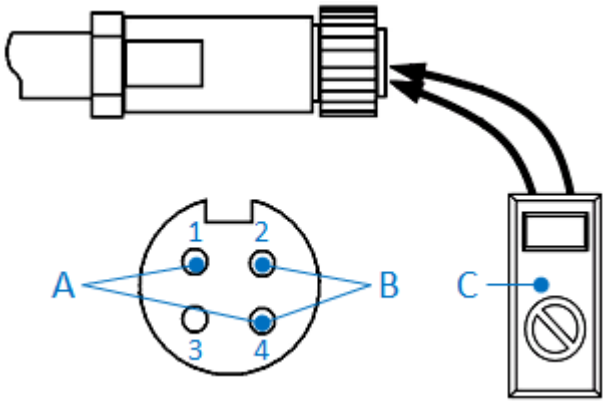
CCD detector has two filaments (1 force-balance filament, 1 catalytic one) of resistance (5±2) Ohm.

Filaments are selected by their resistance value at manufacturing. At manufacturing pair of filaments is assembled together with cable (Sensor assembly).

If one fails to adjust detector's baseline within range 800 – 1000 mV, it's necessary to check state of CCD filaments.

- Remove right side GC cover;
- Disconnect cable from CCD amplifiers slot;
- Measure resistance of filaments as shown in figure below.

If the resistance of the sensors is greater than 7 Ohm, then they should be replaced.



A – filament 1, B – filament 2, C – multimeter

Figure 3.3 – Checking resistance of CCD sensors

Sensor's replacement in CCD (P/N 150-2701)

Sensor replacement procedure is recommended to carry out by trained specialists or Chromatec regional representative.

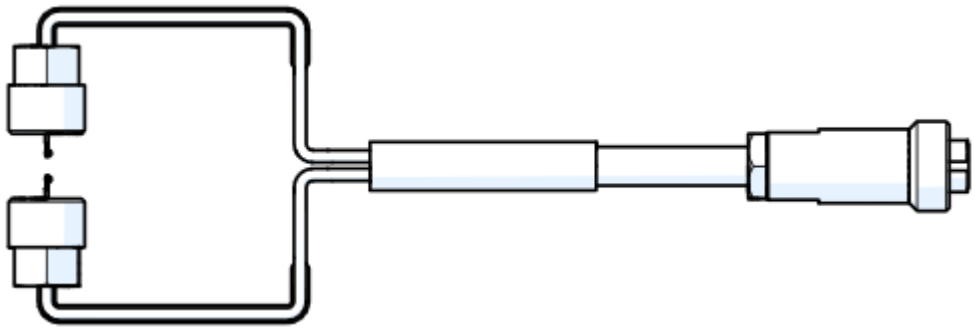
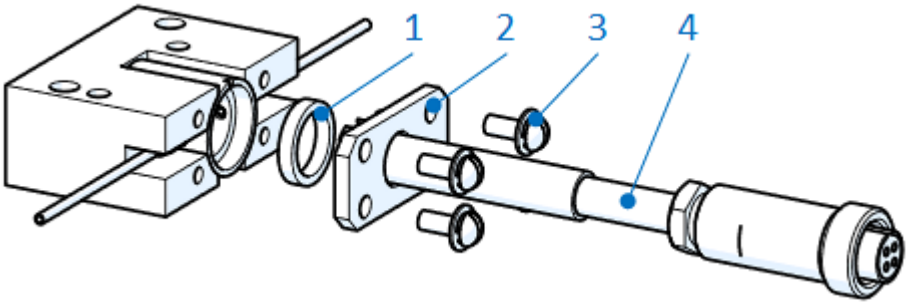


Figure 3.20 – CCD sensor 214.5.863.173

Procedure:

- Disconnect amplifiers sensor from detector;
- Dismount completely all the parts of CCD protective covers and thermal insulation, taking attention on order of its packing; remove cover protecting detectors connections, its dismounting procedure is the same as for TCD;
- Unscrew 2 nuts 214.8.930.312 fixing CCD sensors;
- Carefully take sensors out;
- Thoroughly blow through detectors cell, visually controlling presence of strange particles;
- Place new sensor 214.5.863.173 into the cells, simultaneously checking detector for leaks, tighten nuts 214.8.930.312 with wrench;
- Assemble detector covers and thermal insulation in revers sequence.
-

Sensors replacement in CCD (P/N 150-2711)



1 – O-ring , 2 – pressing plate, 3 – CCD sensor fixing screws , 4 – CCD sensor 5.863.205

Figure 3.21 – CCD sensors replacement

Procedure:

- Disconnect amplifiers sensor from detector;
- Take off detectors covers;
- Remove 4 screws fixing CCD sensors;
- Carefully take sensors out;
- thoroughly blow through detectors cell, visually controlling presence of strange particles;
- Place rubber O-ring into its seat in detectors cell.
- Insert new sensor. Alignment key should be directed vertically upward or downward.

- Tighten 4 screws, pay attention that gap between sensors pressing plate and detectors body must be even.
- Assemble detector in revers sequence.

CCD check for leaks

Check for leaks is carried out once sensors replaced or gas lines mounted.

Procedure:

- Plug column fitting;
- Plug CCD outlet using connector 6.115.120 and plug 8.632.053;
- Set make up gas value at 100 ml/min;
- If flow doesn't go down to 0 in some period of time, it indicates leaks presence. In this case check gas lines connections and tighten screws fixing CCD sensors if necessary;
- If flow went down to 0 ml/min, there is no leak in the detector;
- Remove seals from detectors outlet and fitting in the oven. Unscrewing of seals must be done gradually in order to prevent detectors cell from rapid depressurizing otherwise strong flow can harm filaments.

3.3 Flame Ionization Detector (FID) (P/N 150-2100)

3.3.1 FID operation principle

FID operation is based on change of hydrogen flame background current at entering into it of organic substance.

Background current of hydrogen flame itself is extremely small. Organic substances, burning in hydrogen flame, cause current course, between collector electrode and polarized detector jet. The proceeding current is proportional to quantity of organic substance which has burned down in detector flame.

FID allows to analyze the majority of organic compounds containing fragments C–H. The detector has small response time and small working volume.

Linear range of the FID is over 10^7.

3.3.2 FID construction and installation



Ionizing voltage 200 V is used in FID. It is prohibited to carry out maintenance of the detector at GC being turned on and detector covers being removed.



Hydrogen is used in FID for flame feeding. Hydrogen and air mix is explosive. If detector input fitting in the oven is not properly sealed or column is not properly connected to detector, hydrogen tends to be collected in the oven that may result to explosion.

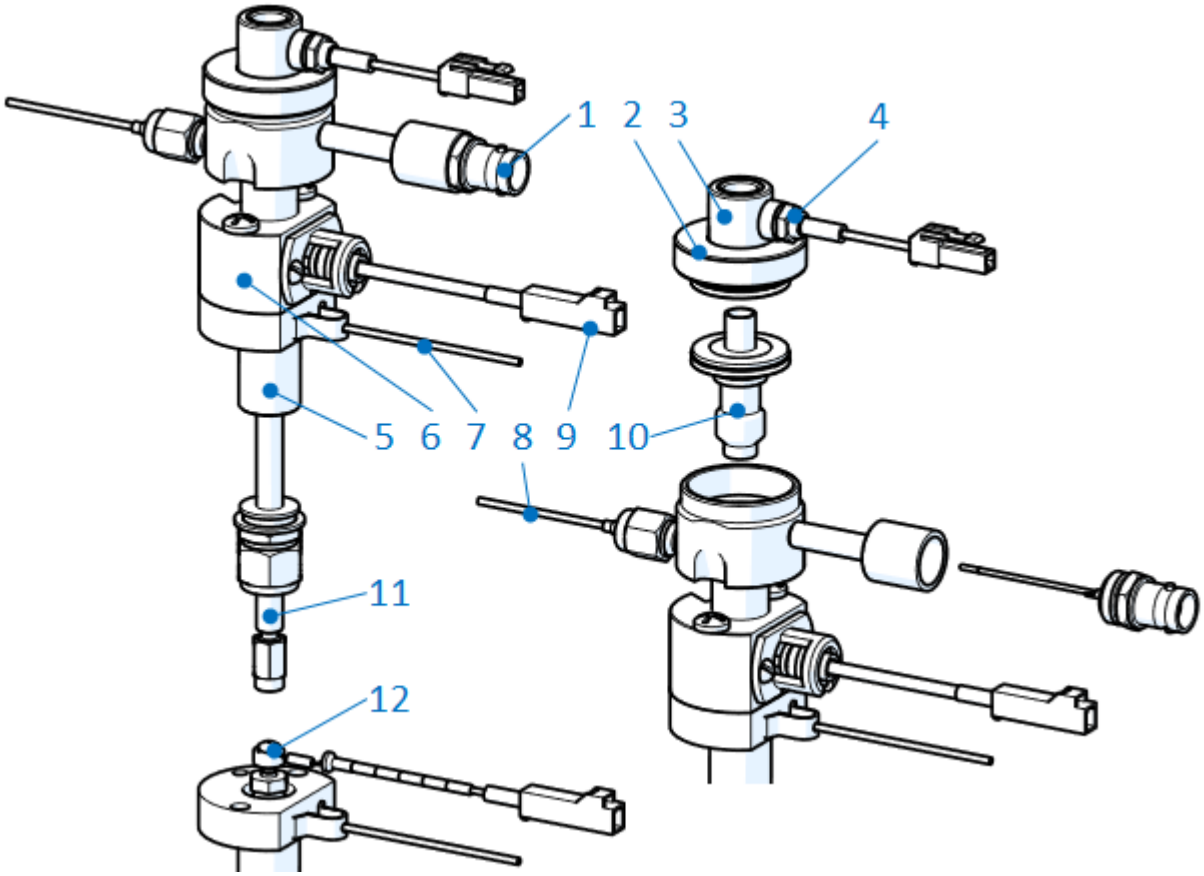


Touching heated detector parts can result in burns.

Design of the FID detector allows you to remove the collector electrode for cleaning without using any tool. Removing time of the collector electrode is no more than 1 minute.

The jets of wider hole (5.877.031–01 and 5.877.031–02) are supplied by order.

FID consists of two parts joint with two brass screws M4.



1 – contact 6.622.051 (to amplifier); 2 – nut 8.936.075, 3 – cap 8.054.257 with an upper insulator (inside), 4 – ignition wire (cable 6.644.182), 5 – body 6.113.220 (bottom part), 6 – body 6.113.161 (top part), 7 – hydrogen supply tube, 8 – air supply tube, 9 – contact 6.622.046 (+200V), 10 – collector electrode 7.660.041 with an bottom insulator, 11 – adapter 6.115.318 (to capillary column, not shown), 12 – jet.

Figure 3.22 – FID construction (P/N 150-2100) in section

On the top part of the detector the following elements are installed:

- Ignition wire;
- Collector;
- Connector with jack to amplifier;
- Connector to “+200 V” stabilizer conducting polarizing voltage to jet.

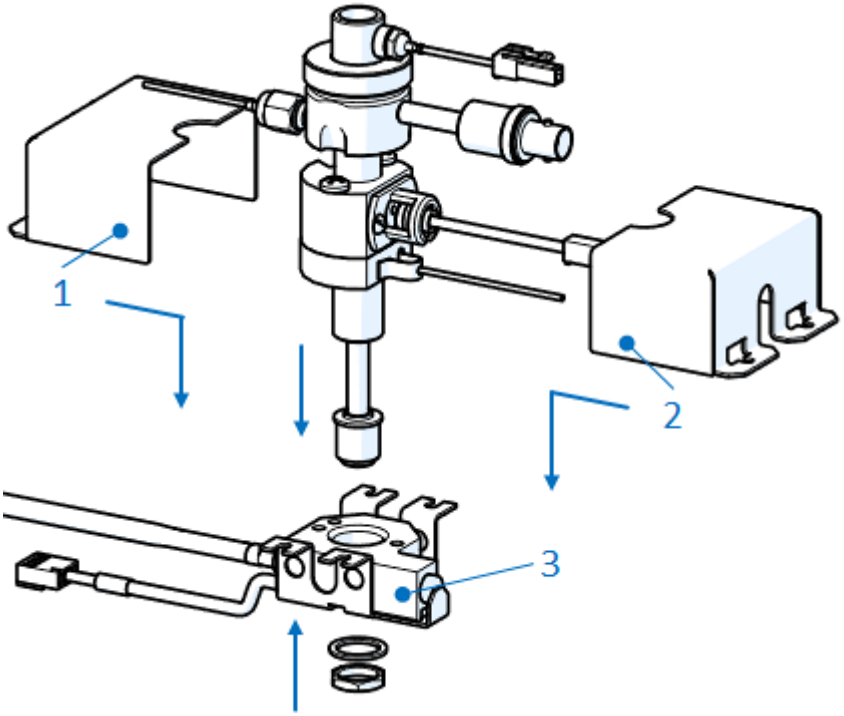
In the bottom part of the detector the following elements are installed:

- FID jet;
- Two rails for top part detector connection.

Jet:

- 5.877.031 hole diameter 0,4 mm;
- 5.877.031-01 (hole 0,5 mm);
- 5.877.031-02 (hole 0,6 mm);

The detector is installed into standard landing place of the heater.



1, 2 – covers, 3 – heater

Figure 3.23 – FID installation

Detector input fitting passes through hole of heater and is tightened in oven by nut. The pin in detector body bottom part enters into heater centering hole. The detector bottom part is closed by covers 1 and 2. Two FID detectors can be installed in the chromatograph on separate heating panels.

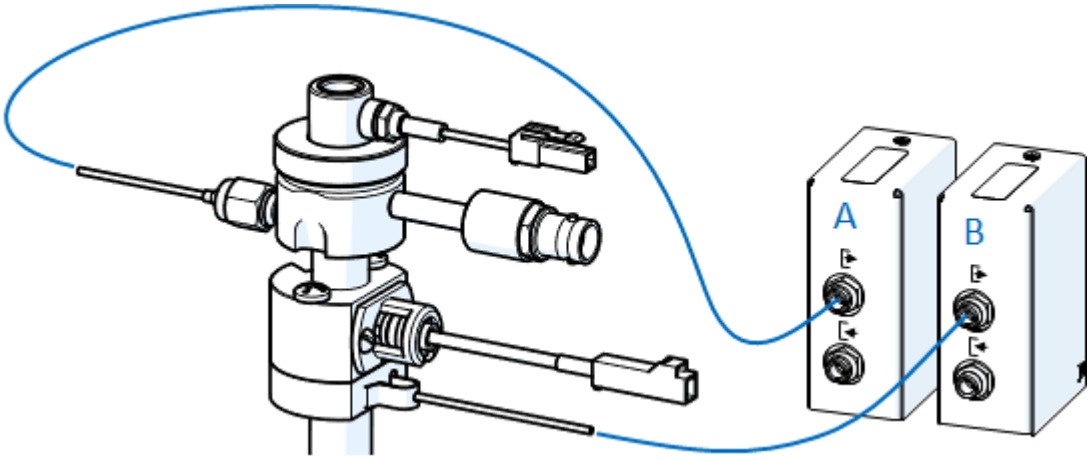
The stabilizer 200V board, supplying polarizing voltage on the jet, is located in the right-side compartment. If two detectors FID installed in the GC, polarizing voltage is connected into the same board.

Amplifier is located in the right-side compartment as well. Cable from amplifier is connected to corresponding detector jack. Detectors number identification (FID-1, FID-2) is provided by amplifier.

Ignition wire is connected to ignition board that is located on the power board in rear side compartment.

3.3.3 FID pneumatic connections

FID gas supplying depending on its Part Number configuration is shown below.

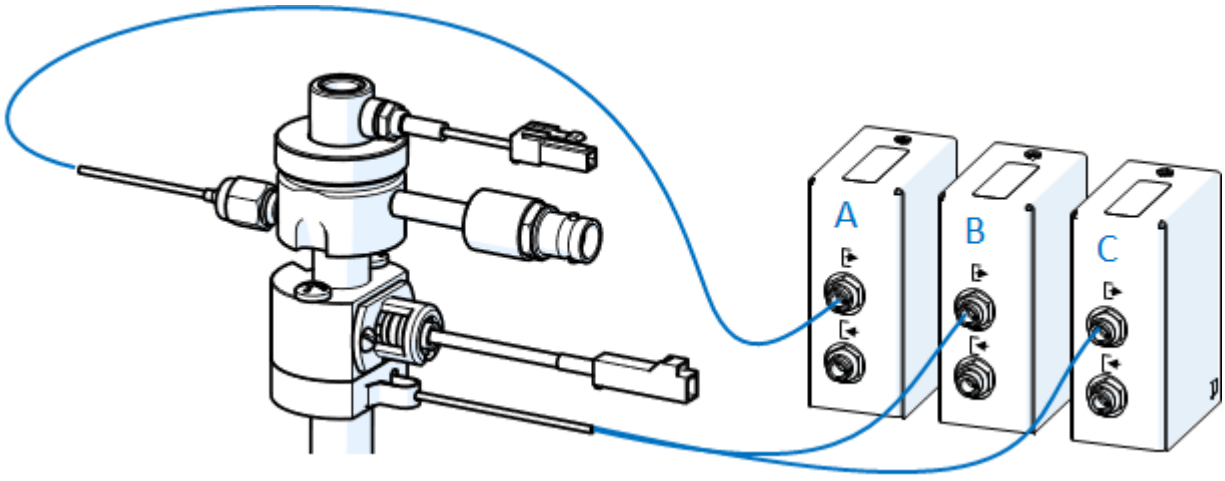


A – DFC-10 air, B – DFC-10 hydrogen

Figure 3.24 – Flow diagram. FID P/N 150-2102 for packed column

When using methanizer, hydrogen flow is connected into Tee-connector before methanizer. FID hydrogen tube is sealed with nut 8.930.167, ferrule 8.658.052, washer 3 and plug 8.632.206 (from shipping kit).

When operating with capillary column, carrier gas makes up flow is added into hydrogen flow.



A – DFC-10 air, B – DFC -10 hydrogen, C –DFC 10 make up gas

Figure 3.25 – Flow diagram. FID P/N 150-2101 for capillary column

Make up flow is connected via tubing ∅1.6 mm into hydrogen tube using T-joint 6.453.094. Gases supply of two flame detectors FID-FID is provided similarly. At the same time, each controller feeds two detectors simultaneously. Gases supply is provided through flow splitters 5.885.010 and 5.885.010-01. A more detailed description of the flow splitters is given in Section 3.4.3 of Part 1 of the UM.

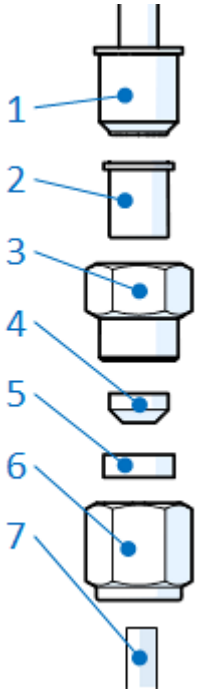
3.3.4 Columns connection to FID

Packed column connection

Rubber ferrules are recommended for use at temperature in the oven less than 250°C.

To connect packed column, install the isert 8.223.507 and the fitting 8.652.307. Tightening force of the fitting 8.652.307 "by hand" followed by turning the key by 10-15 °.

Put column end into insert 8.223.507 to the end. Tightening force of the fitting 8.930.172 "by hand".



1 – FID fitting, 2 – insert 8.223-507, 3 – fitting 8.652.307, 4 – ferrule 8.220.380 (rubber), 5 – sleeve 8.223.013, 6 – nut 8.930.172, 7 – packed column

Figure 3.4 – Packed column connection with rubber ferrules

Instead of ferrule 8.220.380 and sleeve 8.223.013, ferrule 8.453.038 (with graphite) can be used/

Graphite ferrules are allowed to use in the oven up to 450°C. Tightening force of the fitting 8.652.307 "by hand" followed by turning the key by 10-15 °. Place end of the column into insert up to the stop. Hold the column in this position and tighten the nut 8.930.172 finger tight. Use a wrench to tighten the nut at additional 30-60 degrees.

Be careful when tightening glass column with graphite ferrules in order to avoid its breakage.

Capillary column connection

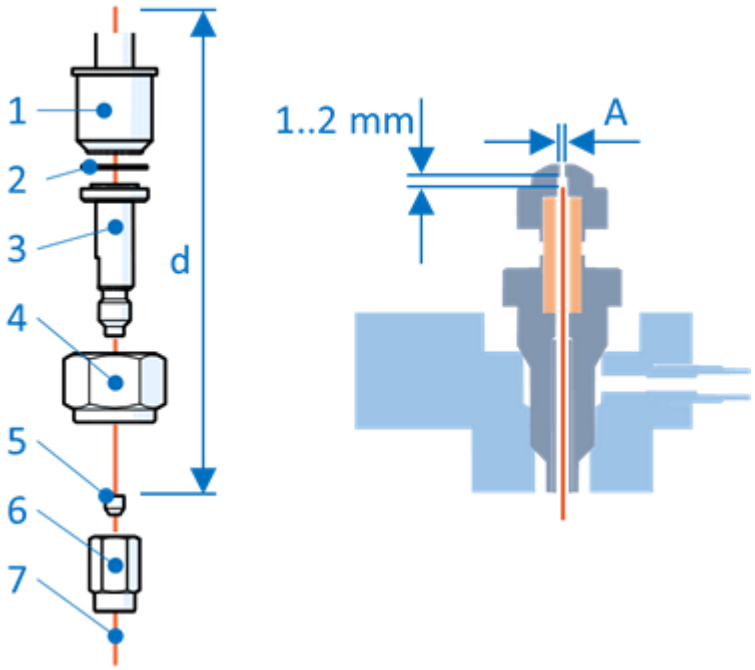
Fit adapter 6.115.318-01 before capillary column installation. Assembly of the adapter is shown on figure below.

Place washer 2 (8.942.247) on the insert 3 (6.278.008) and put it into nut 4 (8.930.268-01) as shown on the figure. Attach this assembly into fitting 1 of detector. Turn by hand, then tighten clockwise by wrench at 10-15°.

Put on nut 6 (8.930.339) and ferrule 5 (e.g., #072626 SGE, with its cone into nut) on the end of column 7.

Cut column end at (5–20 mm) to avoid graphite ingress into column. The cut should be flat without chips and scratches.

Wipe cut end of the column with lint-free tissue wetted in alcohol in order to remove dust and other contaminants.



1 – FID fitting, 2 – washer 8.942.247, 3 – insert 6.278.008, 4 – nut 8.930.268-01, 5 – ferrule, 6 – nut 8.930.339, 7 – capillary column

d = 98 mm (for FID)

Figure 3.5 – Capillary column connection

Locate nut 6 and ferrule 5 at required distance (d = 98mm for FID) from column end. For convenience, use column measuring tool 6.236.249 from shipping kit. Install column end into measuring tool, align column end at mark (5) for FID detector. Then tighten nut 6 by wrench at 60-90 degrees so that column cannot be withdrawn from the fitting (column is tightened by ferrule holder). Then release nut.

Insert the column into detector, tighten nut 6 by hand. Then tighten nut by wrench at 45-60 degrees so that column cannot be withdrawn from the fitting (column is tightened by ferrule holder).

When capillary column installation, it’s necessary to pay attention that column end shall be set at 1 – 2 mm below jet hole. Jet hole A may be 0.4 mm, 0.5 mm or 0.6 mm.

Column installation might be incorrect, if capillary outer diameter less than jet hole diameter and column level in detector jet is adjusted “up to the stop” (distance exceeds 80 mm). Detector usually fails to ignite in this case.

At installation of capillary columns with aluminized external surface it is necessary to pay attention that such column can cause short circuit of polarizing voltage of jet on the chromatograph casing. In this case in the stabilizer 200 V there is restriction of output voltage. Absence of polarizing voltage on jet is shown in FID sensitivity falling. At installation of such column by way “up to the stop and (1-2) mm back”, make sure that "back" moving of column is provided.

3.3.5 FID application features

Basic FID operating condition is steady flame burning. Steady flame burning is provided with detector geometry and ratio of carrier gas, hydrogen and air. It is recommended to keep ratio of hydrogen and air 1:10.

Table 3.3 – Recommended operating gas flow rates

Gas type	**Flow rate, ml/min**			**Admissible value range**
	Optimal at operation	At Ignition state	At performance check	
Make up gas	30	10	30	10 – 50
Hydrogen	20	35	25	20 – 40
Air	200	200	250	200 – 300

At flame burning water is formed. Detector operation at temperature below 150°C is not recommended.

FID flame ignition algorithm:

- Output signal of the detectors is measured.
- Air and hydrogen flows is supplied for each detector of this channel in accordance with configuration settings
- Ignition wires of the detectors of this channel are heated for 8 seconds.
- Ignition wires are switched off.
- Presence of flame is monitored by increasing the background signal of the detector.
- In case of successful ignition, the gas flow rates set via values in instrumental method.

If the increment of the detector output signal is greater than the increment of the threshold value set in the menu, then the ignition is successful, if not, a second attempt is made to ignite with a hydrogen consumption greater by the amount of hydrogen increment. Thus, the chromatograph makes seven attempts to ignite, after which, in the absence of a flame, the "Idle" stage is set and the message "It is impossible to ignite" is displayed.

3.3.6 FID maintenance

Automatic ignition troubleshooting

Automatic ignition of the detector starts once detector is heated. The GC transfers into “Ignition” state, gas flow rates according to this state are set. Then voltage is supplied to ignition wire, characteristic clap occurs indicating ignition of the detector. If ignition was carried out successfully, one can see baseline signal increment 3 – 5 mV or higher. If baseline increment didn’t happen next ignition attempt takes place with higher hydrogen flow rate. Totally seven attempts of ignition are carried out. If all attempts failed, error message appears. In this case set GC mode again and check all steps of ignition state visually.

Table 3.4 – Automatic ignition troubleshooting

Fault description	**Possible cause**
While igniting, wire is not lighted at all or light is too poor.	Ignition wire power circuit or ignition wire itself is broken down. Resistance shall be 0.2-0.3 Ohm for good wire. If resistance exceeds nominal value, replace it.
Wire is lighted brightly in red but clap doesn’t occur, there is now flame*.	Detector gas feeding malfunction. Check that gases are supplied correctly, connections are leak-free column in the oven is connected correctly.
Igniting occurs (clap is heard) – flame presence is identified by mirror *, but no baseline increment.	Amplifier's cable is broken down; amplifier is not connected or broken down. Check amplifiers connection.
Unstable ignition. Igniting occurs (Clap is heard), increment happens, but while gas flow setting back to operating mode, flame gets out.	Different causes: Hydrogen gas lines are not thoroughly blown out (e.g., after hydrogen generator maintenance). Detectors gas lines connected incorrectly. Humid hydrogen. Bake sorbent, change traps. The jet is broken down, clogged or incorrectly installed. Check and replace the jet. Gas flow rates are set incorrectly (hydrogen, air, make up gas, flow through the column). Change gas mode.

* Flame presence may be identified visually by means of any smooth surface (mirror). Take the mirror to detector top vent, if there are appear vapors condensate on the smooth surface, flame is burning. If surface is clear, there is no flame.

Ignition wire replacement

- Disconnect electric wire from cable;
- Unscrew ignition wire
- Place the new one and connect it.

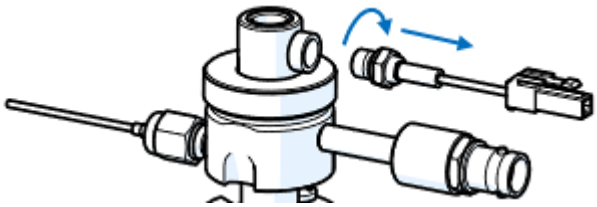


Figure 3.6 – Ignition wire replacement

Collector cleaning

On collector there probably can occur sediments such as white coating of silicon oxide as a combustion product of liquid phase from column, and also as black coating – products of sample combustion. Thus, some decrease of sensitivity and baseline signal noise increase can be observed.

Procedure:

- Disconnect ignition wire cable. Unscrew anticlockwise detectors brass nut. Remove top part of detector.
- Disconnect amplifier. Unscrew side located coaxial contact, then take it out.
- Using pincers or other suitable tool grab collector electrode 7.660.041 together with contact washer 8.940.020, take it out from detector.

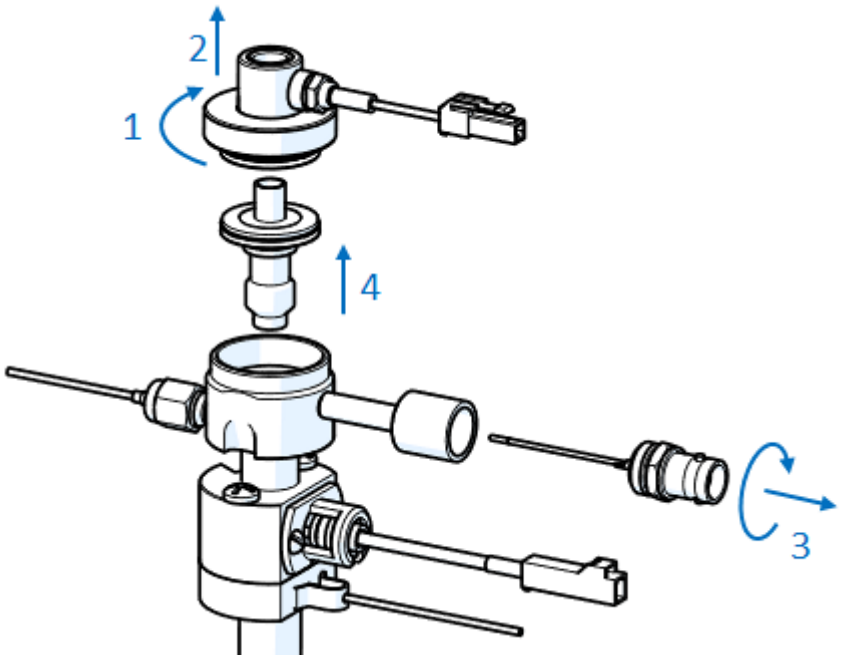


Figure 3.7 Removing the collector electrode

- Opening pincers ends inside bottom insulation, eliminate it from the detector.
- Unscrew ignition wire from detectors top part.
- Eliminate top insulation from detectors top part.

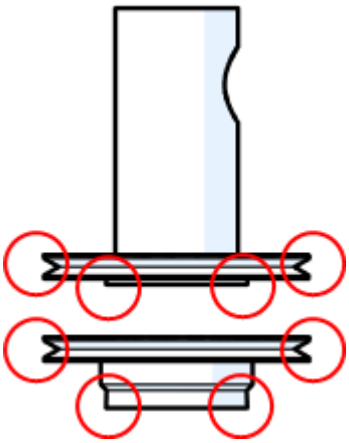
Collector electrode, contact washer and both isolators are recommended to be washed out in acetone or ethyl alcohol. The best effect could be obtained when washing with solvent in ultrasonic bath.

After washing up cleaned parts are to be wiped with dry lint-free tissue and dried at 150 – 200 °C for 15 minutes.

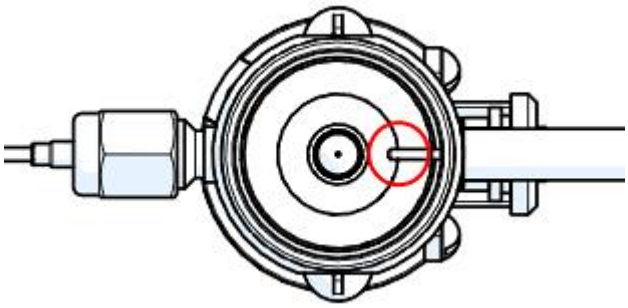
Detector is to be assembled in reverse order. The washed details are recommended to be taken only with pincers.

Detectors assembling features

Before assembling carefully check purity of places on top and bottom insulations indicated in the picture. There must be no contaminants and foreign particles. Otherwise, detectors noise and signal level may exceed the rate significantly.



Once coaxial contact is inserted and amplifier is connected, make sure that coaxial contact tip, lying on the centering electrode, doesn't touch detectors body and juts out in the center of the channel and lies on the protrusion of the collector electrode by about 1 mm.



To prevent electrode tip closing to detectors body when screwing brass nut, it's recommended to keep detectors head from rotating.

Once detector is assembled, this recommended to be heated up to temperature 350 – 380 °C for 1–1.5 hours.

Jet cleaning

In some cases the pollution of the jet with sample products is possible.

At cleaning the jet it is necessary to be careful in order not to damage jet internal surface. Burrs in jet can result in noise increasing.

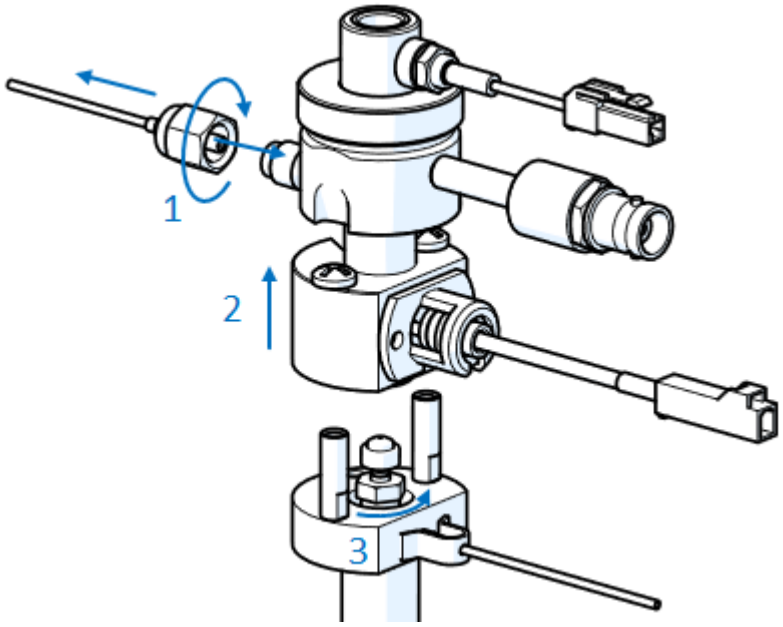
FID jet is quite fragile. At knocking or even at light shifting force to top part of the jet, ceramic insulation may get broken.

Do not take a jet for its top part!

Disassemble and assemble of the jet must be carried out only using flare nut wrench.

Procedure:

- Disconnect all the cables (ignition wire, amplifier, 200V cable).
- Weaken 4 screws fixing two detectors covers. Shift cover outside and remove them.
- Unscrew nut and disconnect air feeding tubing.
- Turn off two screws fixing top detectors body. By light rocking carefully remove top body of the detector.
- Unscrew jet using 8mm flare nut wrench. Be especially careful in order not to break ceramic insulation. To avoid breaking wrench off from hexahedral basis, do push bottom part of the wrench with finger.



- If there are sediment particles in the hole of the jet, it's recommended to clean it with stainless steel wire of 0.3mm diameter. After clearing the jet, it is necessary to blow it with carrier gas or compressed air.
- It is also recommended to use ultrasonic bath with acetone for washing;



- Washed parts should be taken only with pincers;
- Assemble the detector in reverse sequence. The jet is screwed into without force against the stop, then screwed with wrench at 45°.

3.4 Nitrogen phosphorus detector (NPD) (P/N 150-2300)

3.4.1 NPD operation principle

NPD operation is based on that at entering into hydrogen flame of nitrogen phosphorus detector the salts vapors of alkaline metal the ionization degree of phosphorus and nitrogen containing compounds raises.

Source of alkaline metal vapors is the sleeve from the pressed salt cesium bromide (CsBr), located around the jet. During flame burning the sleeve slowly evaporates, and salt vapors get in flame. The analyzed substance, getting in flame of hydrogen jet, decomposes and decomposition products are ionized on mechanisms of specific reactions with participation cesium bromide vapors. The ions which occurred as a result of these reactions under action of voltage detector enclosed to electrodes move to collector electrode, gathered by it and form detector signal.

The detector is intended for selective definition phosphorus and nitrogen containing organic compounds. In comparison with FID, NPD sensitivity for nitrogen containing compounds is higher 50-100 times, for phosphorus containing compounds – 500-1000 times and more.

NPD sensitivity and selectivity to various phosphorus and nitrogen containing compounds is caused by ionization potential of heteroatom decomposition products at these compounds' combustion (Table 3.5).

For standard substances and test conditions, the linear range of NPD is over 10^4 for nitrogen-containing and phosphorus-containing compounds. The selectivity of NPD is not worse than 25000/1 for the ratio of nitrogen to carbon and not worse than 200000/1 for the ratio of phosphorus to carbon by weight.

Table 3.5 – Ionization potentials of some heteroatom compounds

Nitrogen compounds	**Ionization potential, eV**	**Phosphorus compounds**	**Ionization potential, eV**
N2	6,8 / 23,5	P	10,9
NO	9,5	PH3	9,9
NO2	11	PO	0,4
NH3	11,5	PCl3	3,2
N2O	12,9	PH4Cl	1,6
(C6H5)3N	6,8	NP	0,9
		(C6H5)3P	7,4

The less ionization potential of heteroatom decomposition products, the greater signal should be expected from injection in the detector of this compound.

NPD response also depends on molecule structure which heteroatom is in. Therefore, in some cases, at small share of nitrogen or phosphorus atoms in analyzed hydrocarbon compound, for example,

at the analysis of some nitrogen containing alkyl– and aryl– derivative, FID response can be higher, than NPD response.

3.4.2 NPD construction and installation



Ionizing voltage 200 V is used in NPD. It is prohibited to carry out maintenance of the detector at GC being turned on and detector covers being removed.



Hydrogen is used in NPD for flame feeding. Hydrogen and air mix is explosive. If detector input fitting in the oven is not properly sealed or column is not properly connected to detector, hydrogen tends to be collected in the oven that may result to explosion.

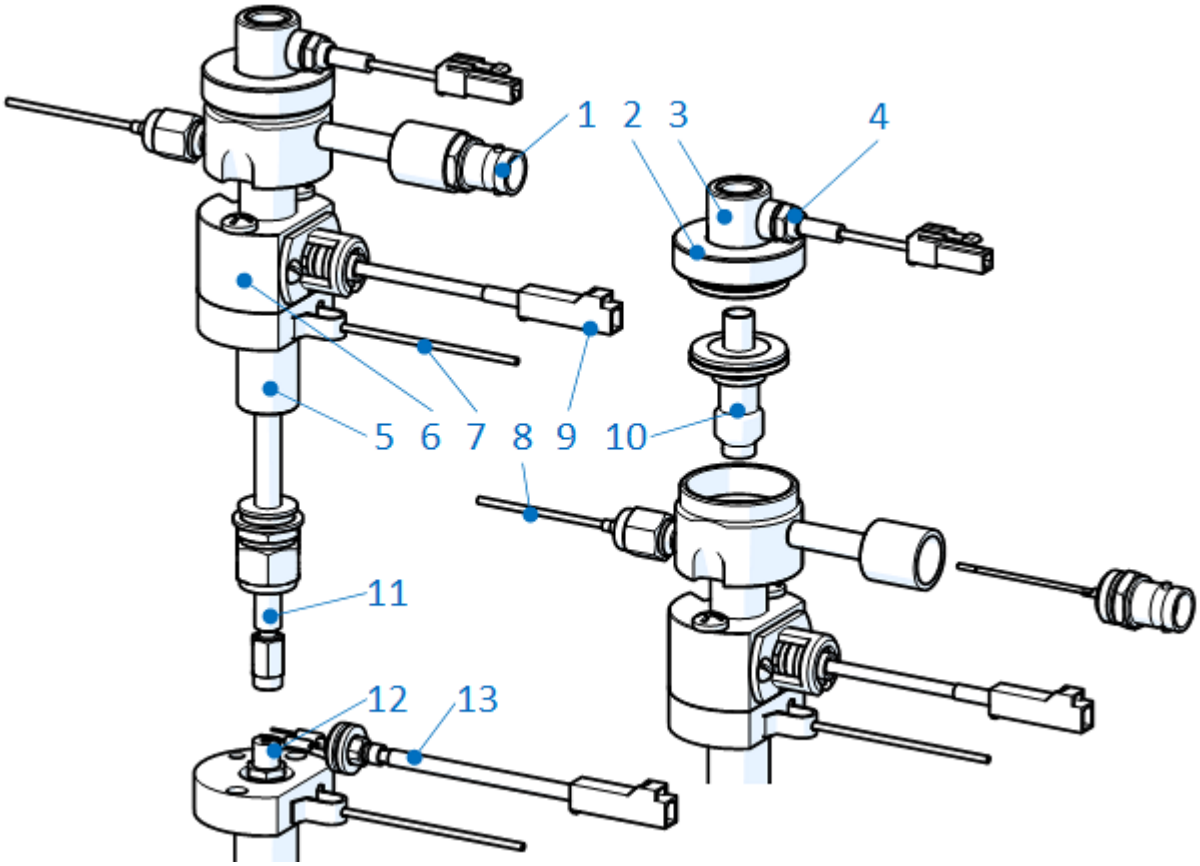


Touching heated detector parts can result in burns.

NPD detector is essentially similar to FID detector

), but differs by salts sleeve serving as a vapors source. Salt sleeve 12, being consisted of pressed cesium bromide (CsBr), is screwed on the jet. Polarizing voltage 200V connected to contact 13, this provides ions movement to collector electrode 10, forming signal of the detector. Signal to amplifier is transferred through the contact 1.

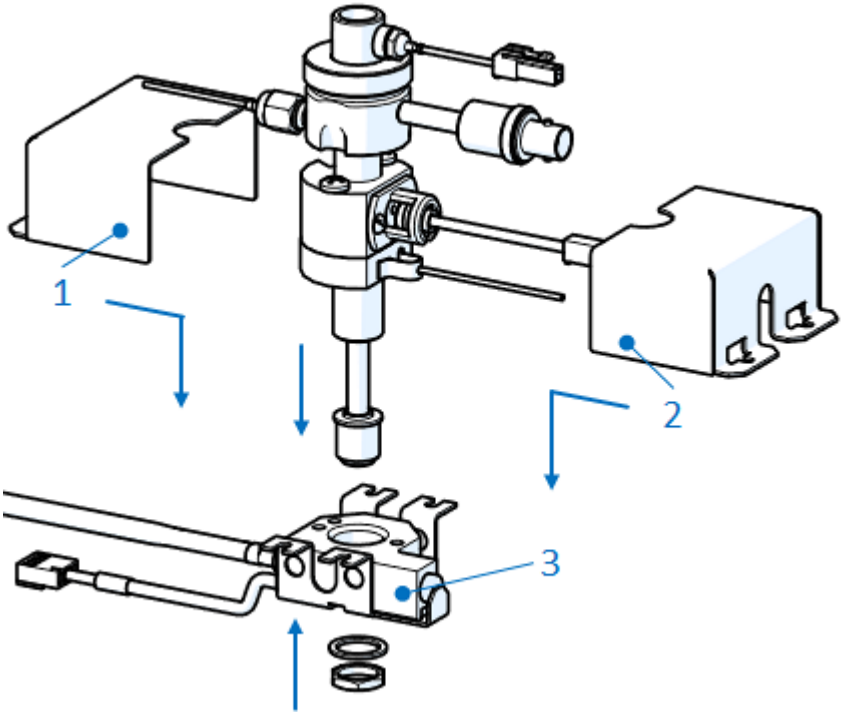
Design of the NPD detector allows you to remove the collector electrode for cleaning without using any tool. Removing time of the collector electrode is no more than 1 minute.



1 – coaxial contact 6.622.051 (to amplifier), 2 – nut 8.936.075, 3 – cap 8.054.257 with upper insulator (inside), 4 – ignition wire (cable 6.644.182), 5 – body 6.113.220 (bottom), 6 – body 6.113.161 (upper, 7 – hydrogen supply tube, 8 – air supply tube, 9 – connector 6.622.046 (+200V), 10 – collector electrode 7.660.041 with bottom insulator, 11 – adapter 6.115.318 (to capillary column, not shown), 12 – sleeve CsBr, 13 – connector 6.622.049 (+200V).

Figure 3.26 – NPD (P/N 150-2300) construction

The detector is installed into standard landing place of the heater (Figure 3.27). Detector input fitting passes through hole of heater and is tightened in oven by washer and nut. The pin in detector body bottom part enters into heater centering hole. The detector bottom part is closed by covers 1 and 2.



1, 2 – covers, 3 – heater

Figure 3.27 – NPD installation

Electrical cables are connected in the same way as for FID detector.

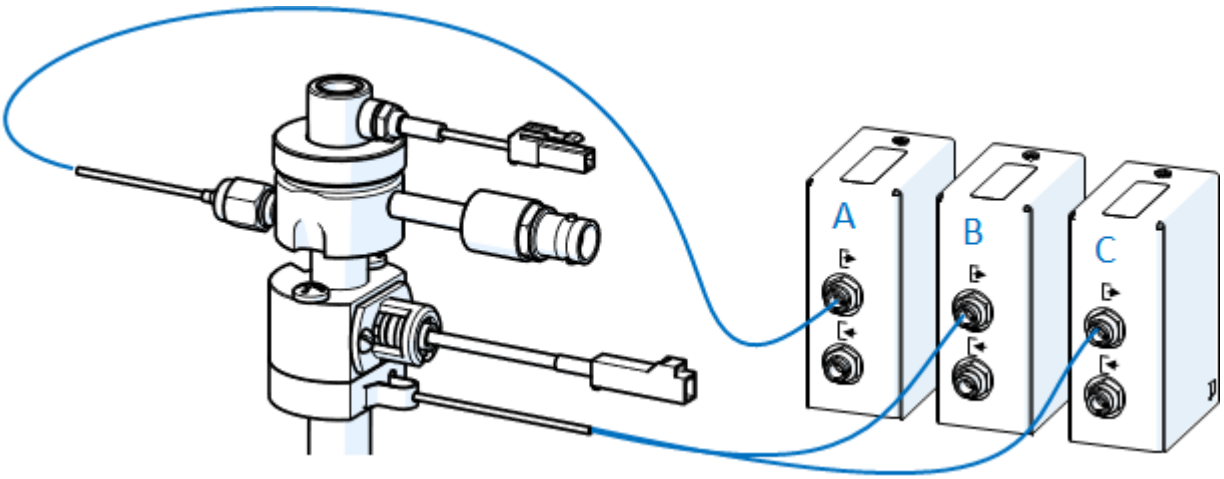
The stabilizer 200V board, supplying polarizing voltage on the jet, is located in the right-side compartment. If two detectors (FID-NPD) installed in the GC, polarizing voltage is connected into the same board.

Amplifier is located in the right-side compartment as well. Cable from amplifier is connected to corresponding detector jack.

Ignition wire is connected to ignition board that is located on the power board in rear side compartment.

3.4.3 NPD pneumatic connections

Gas supply of the NPD detector when working with a capillary and packed column. Nitrogen is used as make up gas.



A – DFC-10 air, B – DFC-10 hydrogene, C – DFC-10 make up gas

Figure 3.28 – Flow diagram FID P/N 150-2301 for capillary column

Make up flow (Nitrogen) is connected via tubing ∅1.6 mm into hydrogen tube using T-joint 6.453.094.

If packed column connected the same pneumatic connection is applied. Male up gas can be set null.

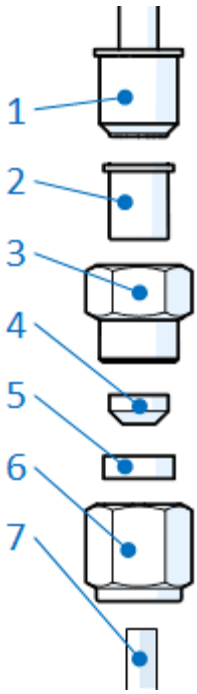
3.4.4 Columns connection to NPD

Packed column connection

Rubber ferrules are recommended for use at temperature in the oven less than 250°C.

To connect packed column, install the isert 8.223.507 and the fitting 8.652.307. Tightening force of the fitting 8.652.307 "by hand" followed by turning the key by 10-15 °.

Put column end into insert 8.223.507 to the end. Tightening force of the fitting 8.930.172 "by hand".



1 – NPD fitting, 2 – insert 8.223-507, 3 – fitting 8.652.307, 4 – ferrule 8.220.380 (rubber), 5 – sleeve 8.223.013, 6 – nut 8.930.172, 7 – packed column

Figure 3.8 – Packed column connection with rubber ferrules

Instead of ferrule 8.220.380 and sleeve 8.223.013, ferrule 8.453.038 (with graphite) can be used/

Graphite ferrules are allowed to use in the oven up to 450°C. Tightening force of the fitting 8.652.307 "by hand" followed by turning the key by 10-15 °. Place end of the column into insert up to the stop. Hold the column in this position and tighten the nut 8.930.172 finger tight. Use a wrench to tighten the nut at additional 30-60 degrees.

Be careful when tightening glass column with graphite ferrules in order to avoid its breakage.

Capillary column connection

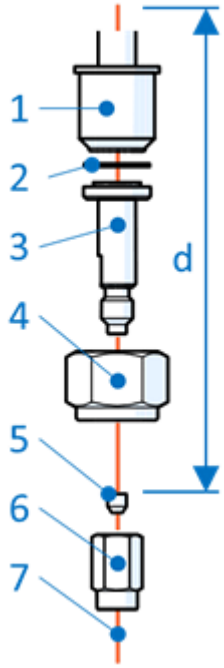
Fit adapter 6.115.318-01 before capillary column installation. Assembly of the adapter is shown on figure below.

Place washer 2 (8.942.247) on the insert 3 (6.278.008) and put it into nut 4 (8.930.268-01) as shown on the figure. Attach this assembly into fitting 1 of detector. Turn by hand, then tighten clockwise by wrench at 10-15°.

Put on nut 6 (8.930.339) and ferrule 5 (e.g., #072626 SGE, with its cone into nut) on the end of column 7.

Cut column end at (5–20 mm) to avoid graphite ingress into column. The cut should be flat without chips and scratches.

Wipe cut end of the column with lint-free tissue wetted in alcohol in order to remove dust and other contaminants.



1 – FID fitting, 2 – washer 8.942.247, 3 – insert 6.278.008, 4 – nut 8.930.268-01, 5 – ferrule, 6 – nut 8.930.339, 7 – capillary column

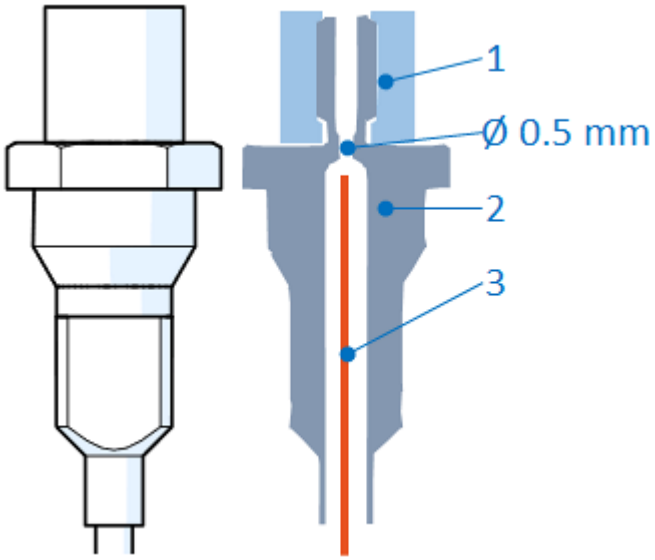
d = 88 mm (for NPD)

Figure 3.9 – Capillary column connection

Locate nut 6 and ferrule 5 at required distance (d = 88mm for NPD) from column end. For convenience, use column measuring tool 6.236.249 from shipping kit. Install column end into measuring tool, align column end at mark (4) for NPD detector. Then tighten nut 6 by wrench at 60-90 degrees so that column cannot be withdrawn from the fitting (column is tightened by ferrule holder). Then release nut.

Insert the column into detector, tighten nut 6 by hand. Then tighten nut by wrench at 45-60 degrees so that column cannot be withdrawn from the fitting (column is tightened by ferrule holder).

When capillary column installation, it's necessary to pay attention that column end shall be set at 1 – 2 mm below NPD jet hole. NPD jet hole diameter is 0.5 mm. Column installation might be incorrect, if capillary outer diameter less than jet hole diameter (0.32mm i.d. or less) and column level in detector jet is adjusted "up to the stop" (distance exceeds 80 mm). Detector usually fails to ignite in this case.



1 – sleeve CsBr, 2 – NPD jet, 3 – capillary column

Figure 3.10 – Capillary column installation details

3.4.5 NPD application features

Sensitivity of the detector depends mainly on salts vapors concentration in flame. Concentration of salts vapors is defined by detector operating mode, basically by detector temperature and hydrogen flow. The higher the detector temperature or bigger hydrogen flow is, the higher detector sensitivity and vice versa.

Concentration of salts vapors depends also on condition of vapors source. In time the geometrical sizes of salt source decrease, quantity of vapors in flame decreases, therefore sensitivity goes down.

Table 3.6 – Recommended NPD gas feeding

Gas type	**Gas flow, ml/min**			**Possible values range**
	Optimum in operation	At ignition	At check	
Carrier gas	30	10	20	10 – 50
Hydrogen	12–14	30	12–14	12 – 18
Air	200	200	130	200 – 300

Recommended value of detector background signal – from 25 to 100 mV at which the maximum ratio of signal/noise is achieved. The level of detector baseline signal depends on hydrogen flow, detector temperature, salt source state. To adjust desirable baseline level, it's recommended to set hydrogen flow rate precisely. Even minor alteration of hydrogen flow (0,1 ml/minutes) can result in essential change of Baseline level. If required baseline level cannot be obtained even at maximum hydrogen level (up to 18 ml/min), salt source is recommended to be changed.

Recommended detector operating temperature is not less than 250°C.

Detector temperature and hydrogen flow set values have an influence on selectivity and sensitivity of analyzed nitrogen and phosphorus containing organic compounds.

To obtain greater sensitivity for nitrogen containing compounds, use detector high temperature (330-350 °C) in combination with small hydrogen flow.

At phosphorus containing compounds determination, detector low temperature (250-270 °C) with higher hydrogen flow rate is preferable.

NPD flame ignition algorithm:

- Output signal of the detectors is measured.
- Air and hydrogen flows is supplied for each detector of this channel in accordance with configuration settings
- Ignition wires of the detectors of this channel are heated for 8 seconds.
- Ignition wires are switched off.
- Presence of flame is monitored by increasing the background signal of the detector.
- In case of successful ignition, the gas flow rates set via values in instrumental method.

If the increment of the detector output signal is greater than the increment of the threshold value set in the menu, then the ignition is successful, if not, a second attempt is made to ignite with a hydrogen consumption greater by the amount of hydrogen increment. Thus, the chromatograph makes seven attempts to ignite, after which, in the absence of a flame, the "Idle" stage is set and the message "It is impossible to ignite" is displayed.

3.4.6 NPD maintenance

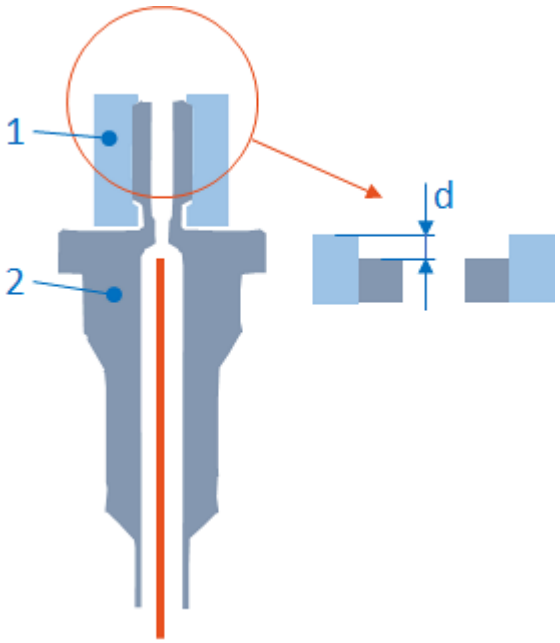
Procedures on automatic ignition troubleshooting, collector cleaning are the same as for FID detector (see section 3.3.6).

Specific points at unstable automatic ignition of NPD detector may appear:

- Salt source being incorrectly installed or worn out,
- Too low hydrogen flow rate.

Salt vapor source renewal

NPD Detector is mechanically similar to FID detector, but differs from it by presence of salt vapors source, representing the sleeve from cesium bromide, screwed on the jet (Figure 3.29).



1 – Salt sleeve (CsBr) 8.223.040, 2 – jet 5.877.010, d = 0.4±0.1mm

Figure 3.29 – NPD jet with salt tip

Salt vapors source service life depends on flame burning modes. High operating hydrogen flow rate results in source service life shortening.

If after detector long work the level of background signal has considerably fallen and hydrogen flow adjustment to the maximum possible value (up to 18 ml/min, see table 3.6) doesn't lead to desirable baseline level, the reason for it can be change of salt vapors source geometrical sizes. It is observed in that the working surface of salt sleeve becomes lower than jet edge. In this case, to recover baseline level, slight unscrewing of salt sleeve from the jet support can be recommended, so that working surface of salt sleeve gets protruded comparatively the jet cut slightly (interim measure). Another way, replacement of salt source with installing new one is recommended.

Procedures for NPD jet taking off and installations with the salt sleeve are similar to procedures of FID jet replacement.

The salt sleeve is fragile, easily breaks, therefore removal and installation of the salt sleeve should be carried out careful in order to not damage it and that scraps of the sleeve would not get inside jet.

In first working hours with the new sleeve there is an intensive evaporation from sleeve surface, the big drift of background signal will be observed therefore. During this time the sleeve surface will slightly fuse by hydrogen flame. Then process of evaporation (accordingly and background signal drift), will stabilize.

In first operating time with vapors new source minimum possible hydrogen flow – approximately 10 ml/min is recommended.

Procedures:

- Take the jet off in the same way as with FID jet;
- Remove used salt sleeve;
- Wash out the jet in acetone (washing in ultrasonic bath is desirable);
- Install the new salt sleeve (see instructions below);
- After washing, the parts should be taken only with pincers;
- Assemble the detector in reverse order;
- Screw the jet in without force against the stop, then tighten with wrench at 45°.

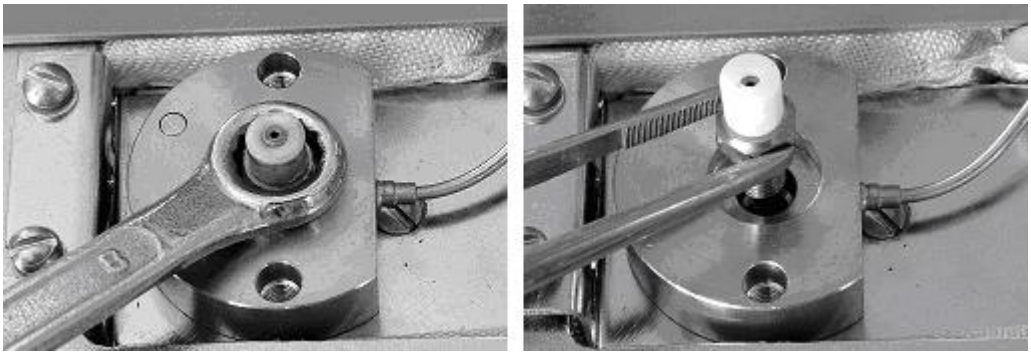


Figure 3.11 – Salt sleeve replacing

New salt sleeve installation

Procedures:

- Inspect new sleeve. The working surface from the hole side of smaller diameter should be smooth, flat without dints and heights. The sleeve should be solid, without cracks and chips.
- The sleeve length should be (5.7±0.1) mm. If the sleeve length is longer, it is necessary to grind off sleeve surface from the hole side of bigger diameter using fine-grained abrasive cloth. During handling it is necessary to control perpendicularity of grounded surface to sleeve axis, and to avoid chips.
- Screw the sleeve on the jet with working surface outside. It is necessary to avoid getting of salt chips inside the jet for what during or after accomplishment of this procedure the jet should be blown with compressed air.
- Examine sleeve position on the jet. The distance between jet top point and sleeve top working surface should be approximately (0.2±0.1) mm.



1 – working surface, 2 – surface, subject to grinding

Figure 3.12 – Salt sleeve

When you turn on NPD detector first time with a new salt sleeve, it is recommended to bake out the salt sleeve, which occurs automatically and is implemented in the Chromatec Analytic Software, section 7.5 – Service operations. NPD Sault Source baking.

3.5 Flame Photometric Detector (FPD) (P/N 150-2200)

3.5.1 FPD operation principle

Operation of FPD detector is based on excitation of analyzed molecules in the flame enriched with hydrogen and registration of photons of certain wave length when molecules convert into basic state.

The detector is selective to sulfur containing compounds (394 nm) and phosphorus containing compounds (526 nm).

Detector operation principle can be described in four stages.

For sulfur containing compounds:

- In the first stage decomposition of initial sulfur containing compounds occurs in hot area of hydrogen flame resulting formation of sulfur atoms or hydrogen sulfide;
- In the second stage various convertible reactions in the flame top part with formation of compound S_2 take place;
- On the third one – there is sulfur molecules excitation;
- On the fourth – the excited sulfur molecules coming in flame cold external cone transfer into basic condition, emit light in wide range of waves lengths (300-450) nm.

For phosphorus compounds the processes are similar. Light emission occurs in narrow wave range with maximum of intensity 526 nm.

For standard substances and test conditions, the linear range of FPD is over 10^3 for sulfur-containing and is over 10^4 phosphorus-containing compounds. The selectivity of FPD is not worse than $10^6/1$ for the ratio of sulfur or phosphorus to carbon by weight.

3.5.2 FPD construction and installation

Detector (Figure 3.) allows using FPD in single or dual flame modes. These modes are described at length in section 3.5.5.

To prevent PMT failure, the detector can be disassembled only when the GC switched off.



In FPD the voltage 800 V for PMT supply is used. It is necessary to follow safety measures at detector installation.



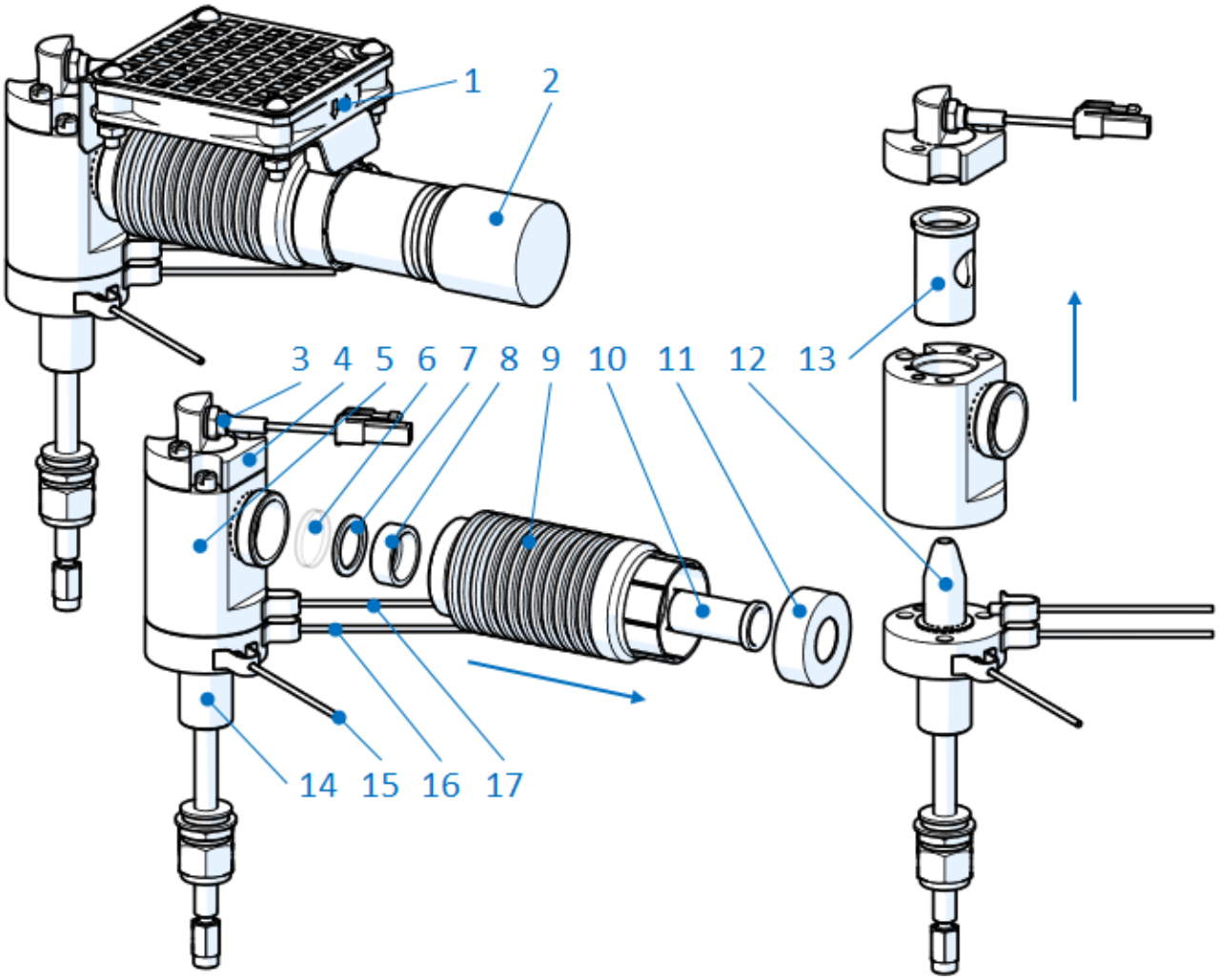
In FPD hydrogen for flame feeding is used. At hydrogen supplying it is necessary to pay attention to possibility of hydrogen leaks into oven when detector column fitting is not sealed or column is not connected.

While FPD operating, flame particularly burns out of detector chamber at the outlet of working chamber (Figure 3., pos.5). This flame is not visible by naked eye. Such a feature is not damage. Keep closed top cover of the GC while working with FPD.

While FPD operating follow fire safety measures GOST 12.1.004, as well as measures developed by customer according to GOST 12.1.007.



At detector high temperatures (over 300 °C) it is necessary to pay attention that the detector top part can be heated to temperature which can cause burns.



1 – cooler 5.883.001; 2 – photoelectron multiplier – PMT (sensor 5.132.026); 3 – ignition wire (cable 6.644.182; 4 – cap (body 6.113.090); 5 – basic body (body 6.113.088); 6 – disk 8.260.084; 7 – washer 8.942.092; 8 – sleeve; 9 – PMT radiator (body 8.034.342); 10 – light pipe (tube 8.236.194); 11 – light filter 5.886.100-01; 12 – top jet 7.064.002; bottom jet 5.877.011; 13 – chamber 7.015.003; 14 – bottom body 6.113.029; 15 – bottom tubing; 16 – middle tubing; 17 – top tubing.

Figure 3.43 – FPD construction (P/N 150-2200)

FPD working chamber is supplied in two versions – made of stainless steel (7.015.003) and aluminum (7.015.003-01).

FPD light filters are supplied in 2 modifications:

- For sulfur channel (violet, black) with wavelength 394 nm.
- For phosphorus channel (yellow) with wavelength 526 nm.

Light filter 5.886.100-01 is installed in the detector. Filter 5.886.100 is supplied with a FPD detector (P, S)

One of the filters is installed into detector at delivery, another one is packed in shipping kit.

Light filter may be installed into detector with its any side to working chamber.

There is an option for FPD – detector FPD with FI-sensor (Figure 3.30).

Detector FPD with FI-sensor is complete FPD equipped with collector electrode, similar to one used in FID detector. This collector electrode allows carrying out the qualitative analysis of hydrocarbon matrixes on FID channel simultaneously with analysis of phosphorus or sulfur containing compounds on FPD channel.

At operating FPD with FI-sensor, FID amplifier 5.173.029–03 is to be used.

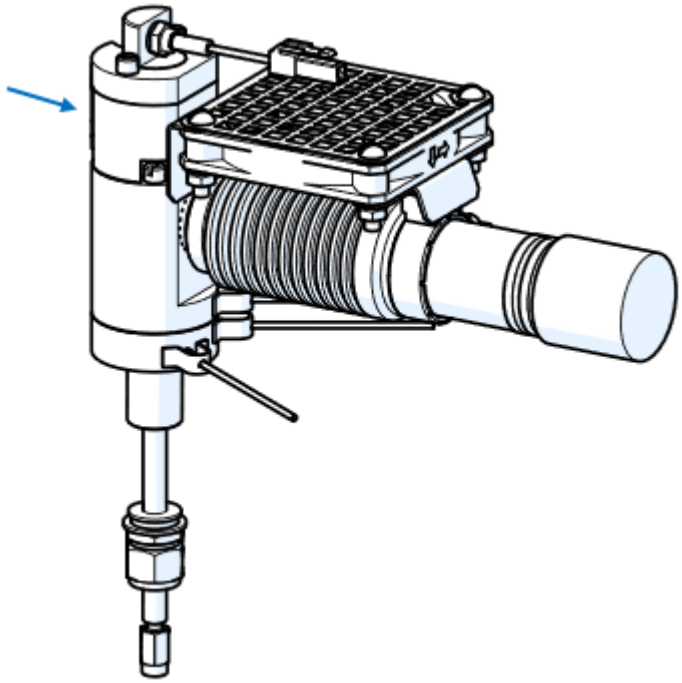
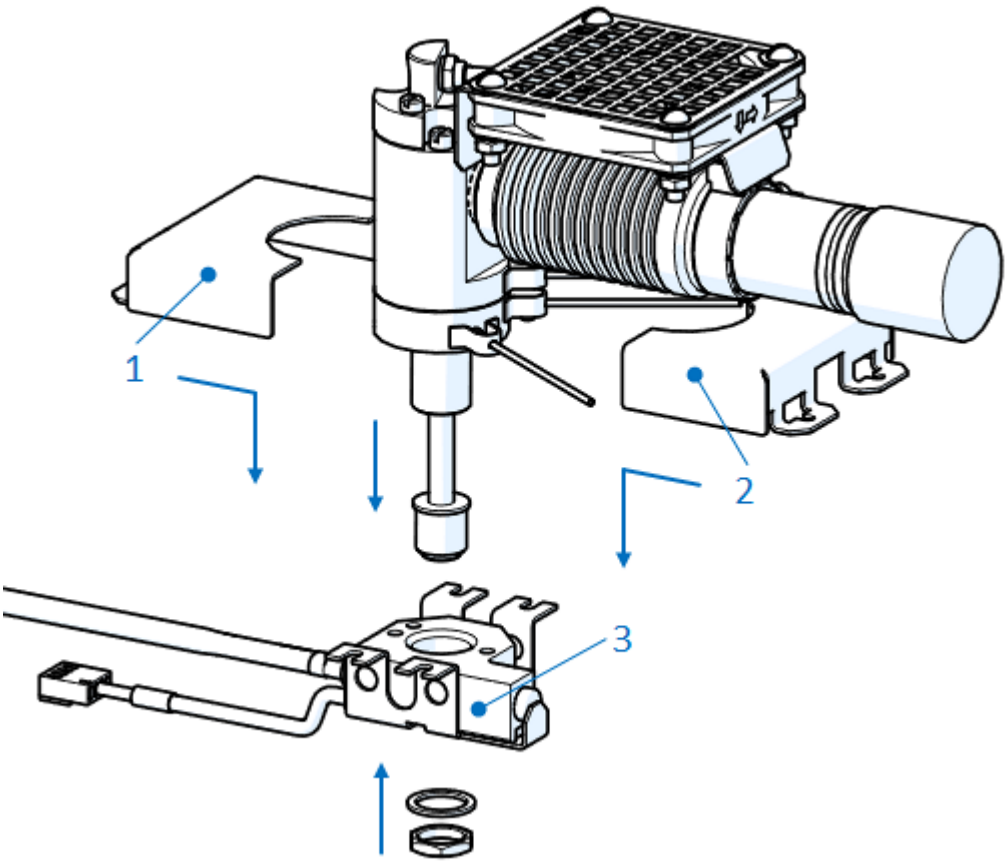


Figure 3.30 – Detector FPD with FI sensor construction

The detector is installed in standard seat. The pin in detector body bottom part enters into centering hole in heater.

Detector column fitting passes through the hole in the heater and is tightened by nut in oven (nut 8.930.137-03, not shown in the figure). The detector bottom part is closed by covers 1 and 2.

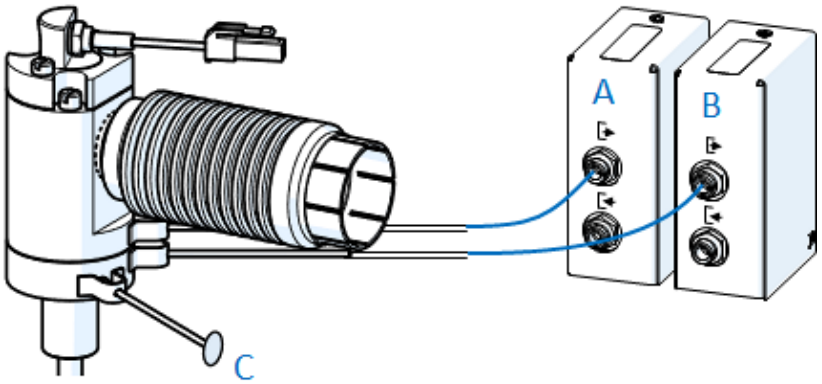


1,2 – Covers, 3 – heater

Figure 3.45 – FPD installation

The stabilizer 800 V is installed in right side compartment. It is intended for PMT supply (see UM pt.1).

3.5.3 FPD pneumatic connections



A – DFC-10, hydrogen “top”, B – DFC-10, air “middle”, C – plug “bottom”

Figure 3.46 – FPD feeding gases in single flame mode

Tubing marking "Top", "Bottom", "Middle" corresponds to their position in FPD casing at vertical.

In single flame mode the bottom pipeline is plugged.

When dual flame mode is required, top and bottom pipelines are to be connected to air, middle pipeline is to be connected to hydrogen.

3.5.4 Columns connection to FPD

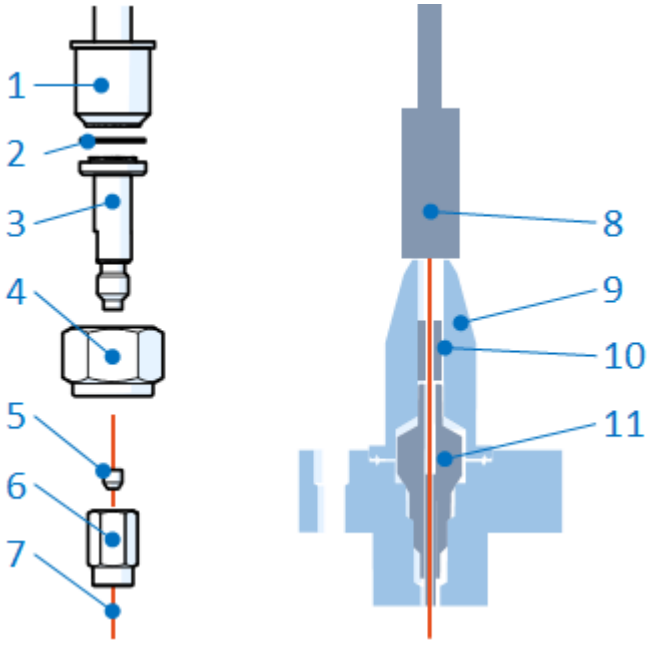
Before column installation into FPD, power off the GC, remove FPD detector top cap

Capillary column connection

Fit adapter 6.115.318-01 before capillary column installation. Assembly of the adapter is shown on figure 3.47.

Place washer 2 (8.942.247) on the insert 3 (6.278.008) and put it into nut 4 (8.930.268-01) as shown on the figure. Attach this assembly into fitting 1 of detector. Turn by hand, then tighten clockwise by wrench at 10-15°.

Cut column end at (5–20 mm) to avoid graphite ingress into column. The cut should be flat without chips and scratches. Wipe cut end of the column with lint-free tissue wetted in alcohol in order to remove dust and other contaminants.



1 – Detector fitting, 2 – washer 8.942.247, 3 – insert 6.278.008, 4 – nut 8.930.268-01, 5 – ferrule, 6 – nut 8.930.339, 7 – capillary column, 8 – stop 8.366.064, 9 – top jet, 10 – centering insert, 11 – bottom jet

Figure 3.13 – – Installation of capillary column

Put on nut 6 (8.930.339) and ferrule 5 (e.g. #072626 SGE, with its cone into nut) on the end of column 7.

Put the stop 8.366.064 into detector cell on the jet as shown below. For single flame mode, the capillary column shall be installed up to the stop of top jet edge precisely.

Locate nut 6 and ferrule 5 at some distance (e.g., ≈ 90mm) from column end. Insert the column into detector until the column end touches the stop 8.366.064. Then fix position of the column and tighten nut 6 by hand.

Tighten nut 6 by wrench at 60-90 degrees so that column cannot be withdrawn from the fitting (column is tightened by ferrule holder).

Put on back the top cap of the detector after column installation terminated. Fix the cap with screws. When you turn on FPD detector first time with a new column cut, it is recommended to anneal the column, which occurs automatically and is implemented in the Chromatec Analytic Software, section 7.5 – Service operations. “Annealing of the column end in the FPD”.

At repeat installation of the column, its annealed end is necessary to cut off not less than (5-10) mm.

Packed column connection for single flame mode

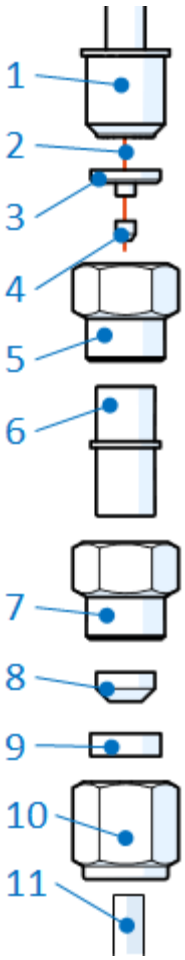
Mounting kit for packed columns connection is optional (available by separate order).

- 214.4.069.026 Mounting kit for packed column with 3.2mm (1/8 ") ends;
- 214.4.069.027 Mounting kit for packed column with 5.4mm ends

214.4.069.027 Mounting kit for colums 5.4 mm is demonstrated on the figure below. The mounting kit 214.4.069.026 has a similar content.

Installation of capillary and column fittings for operation in single flame mode is made in the following sequence.

- Before installation of capillary remove the detector top cap (chromatograph must be switched off).
- Fix the guide 3 (8.224.073) with fitting 5 (8.652.307) on detector fitting. Tighten "by hand", then swage by wrench on (10-15) degrees.
- Insert the ferrule 4 on the capillary 2 (7.352.085) with cone downward as it is shown on the figure. Insert the capillary into the detector, then install the connecting insert 6 (8.224.075) with the cone-shaped socket under the ferrule upward and fit with fitting 7 (8.652.307). At installation and fastening of capillary it is necessary to be careful in order to prevent its breakage. The fitting 6 should be turned by hand.
- With the help of the stop 8.366.064 adjust level of installation of capillary in detector jet and with the help of tweezers install centering insert (figure 3.47).
- Close the top cover of detector.
- Finally swage the fitting 7 by wrench on (10-15) degrees.



1 – Detector fitting, 2 – 214.7.352.085 Capillary (100mm), 3 – 214.8.224.073 Guide, 4 – Ferrule 0.8 mm ID #072626 (SGE), 5 – 214.8.652.307 Fitting, 6 – 214.8.224.075 Insert, 7 – 214.8.652.307 Fitting, 8 – 214.8.220.380 Ferrule (rubber), 9 – 214. 8.223.013 Sleeve, 10 – 214.8.930.172 Nut, 11 – Packed column end 5.4mm OD

Figure 3.31 – Packed column installation in single flame mode

Level of FPD fitting location in single flame mode is below usual fittings. Therefore, the second end of column also should be installed with the help of special lengthened fitting for inlet 6.454.092 (from the detector FPD shipping kit).

Packed column is inserted into guide up to the stop.

At using graphite ferrules 6.453.038 as a sealing, force of nut 8.930.172 tightening "by hand" with the subsequent turning by wrench on (30-60) degrees. At sealing of glass column with graphite ferrules it is necessary to be careful in order to prevent its breakage.

At using rubber ferrules as a sealing, the nut 8.930.172, insert 8.223.013, ferrule 8.220.380 are put on. The force of nut tightening "by hand".

3.5.5 FPD application features

When analyzing hydrocarbon matrixes in single flame mode FPD, if target sulfur containing compounds are not separated from hydrocarbons, quenching flame effect occurs. It results in decrease of sensitivity to analytes due to process of capture of photon radiated by the excited molecule of analyzed sulfur substance. For elimination of such processes dual flame FPD operating mode is recommended. But at work in dual flame mode there is some decrease of FPD sensitivity.

To obtain the best sensitivity and calibration linearity at sulfur containing compounds determination, feeding gases (hydrogen and air) must contain minimum concentrations of hydrocarbons and carbon dioxide, since these impurities cause permanent quenching flame effect.

Particularly, when hydrogen generator and air compressor are used, additional filters are recommended.

Table 3.7 – FPD operating modes

Operation mode	**Column**	**Features**
Single flame	Capillary	Good selectivity, high sensitivity. For analysis of complex hydrocarbon matrixes. Detailed high-quality identification of analyzed components.
Single flame	Packed	Moderate selectivity, high sensitivity. The effect of sensitivity decreases on (30–50) % at overlaying of hydrocarbon peaks.
Dual flame	Capillary	Good selectivity, moderate sensitivity. For analysis of complex hydrocarbon matrixes when maximum sensitivity is not required.
Dual flame	Packed	Good selectivity, moderate sensitivity.

At analysis of hydrocarbon matrixes on FPD it is recommended:

- Using capillary column in single flame mode;
- Using packed column in dual flame mode.

Table 3.8 – Recommended gases flows

Flow, ml/min	**Operation mode**		
	With packed column	With capillary column	Ignition mode
Single flame mode:			
Hydrogen	140 (50*)	140 (50*)	80
Air	40 (50*)	40 (50*)	180
Dual flame mode:			
Top air	170	170	170
Hydrogen	140	140	140
Bottom air	80	40	80

* Mode of maximum sensitivity to phosphorus and sulfur containing compounds (selectivity of the detector is lowered). It is supposed to set the hydrogen and air flow in range of the specified values (from 50 to 140 ml/minutes for hydrogen and 40 to 50 ml/minutes for air). At increase of hydrogen flow and/or decrease of air flow the FPD sensitivity is reduced, selectivity raises.

Detector temperature depends on application (temperature value recommended at 20 °C above maximum column temperature, but not lower than 150 °C; at increasing detector temperature, the response on sulfur goes down).

Two types of working chambers can be used in the detector:

- Stainless steel working chamber (7.015.003). Basic advantage is resistivity to oxidation if there is aqueous condensate in the chamber;
- Aluminum working chamber (7.015.003–01). Basic advantage is higher sensitivity (at 1,5–2 times, than for chamber 7.015.003) thanks to better light-reflecting ability of the chamber. Lack is that aluminum is subject to oxidation under condensate affect.

To prevent condensation in working chamber, it's recommended to set hydrogen flow at cooling at least 50 ml/min).

FPD flame ignition:

In contrast to the ignition algorithm for FID and NPD, the GC here does not constantly monitor the flame, but performs a periodic check of its presence. The program considers that when switching from the "Zero" stage to the "Preparation" stage, the flame does not burn and ignition should be performed in any case. signal measurement at this point is not performed.

Background measurement and flame detection using a method traditional for FID and NPD is not relevant due to the possible variable dark background of the PMT, which makes it difficult to unambiguously determine the presence of flame.

The following algorithm of flame ignition is implemented for FPD:

- The air and hydrogen flow rate specified in the configuration is generated.
- The ignition wire is heated for the current detectors channel for 8 seconds.
- Ignition wire heating switches off.
- Detectors gas flows set at the operating mode.
- 10 seconds after the flow of air and hydrogen to the specified operating mode, the output signal of the detector is measured.
- The air flow rate increases by 20 ml/min relative to the value in the mode (for example, at a given flow rate of 40 ml/min, the value will be set to 60 ml/min).
- After 5 seconds, the detector output is measured.
- If the signal has exceeded the threshold set in the configuration, the flame is considered to be on. Gas flow rates are set in accordance with the values set in the mode.
- Otherwise, the **air flow** increases by the amount specified in the configuration, and the ignition attempt is repeated.

Seven ignition attempts can be made, after which, in the absence of flame, the “Zero” stage is set and the message “Ignition failure” is displayed.

Ignition of a flame in FPD is accompanied by one loud pop. If several frequent pops are heard, this indicates that there is not enough air. The air flow should be increased by 20 mi/min.

3.5.6 FPD maintenance

Troubleshooting on automatic ignition malfunction, collectors cleaning is similar to the same procedures for FID (see chapter 3.3.6).

The causes of unstable ignition specific to the detector may be:

- Damaged or incorrectly installed capillary (column) in the detector,
- Upper part of the detector is too cold (due to forced cooling, for example in the air flow of an air conditioner), multiple pops are heard due to condensation.

For amplification of luminous flux in FPD the specular surface of working chamber is used. While in service the chamber surface and safety glass are dirtied with combustion products and cloud. Reflecting characteristics of chamber and carrying capacity of safety glass decrease that result in decrease of detector desired signal.

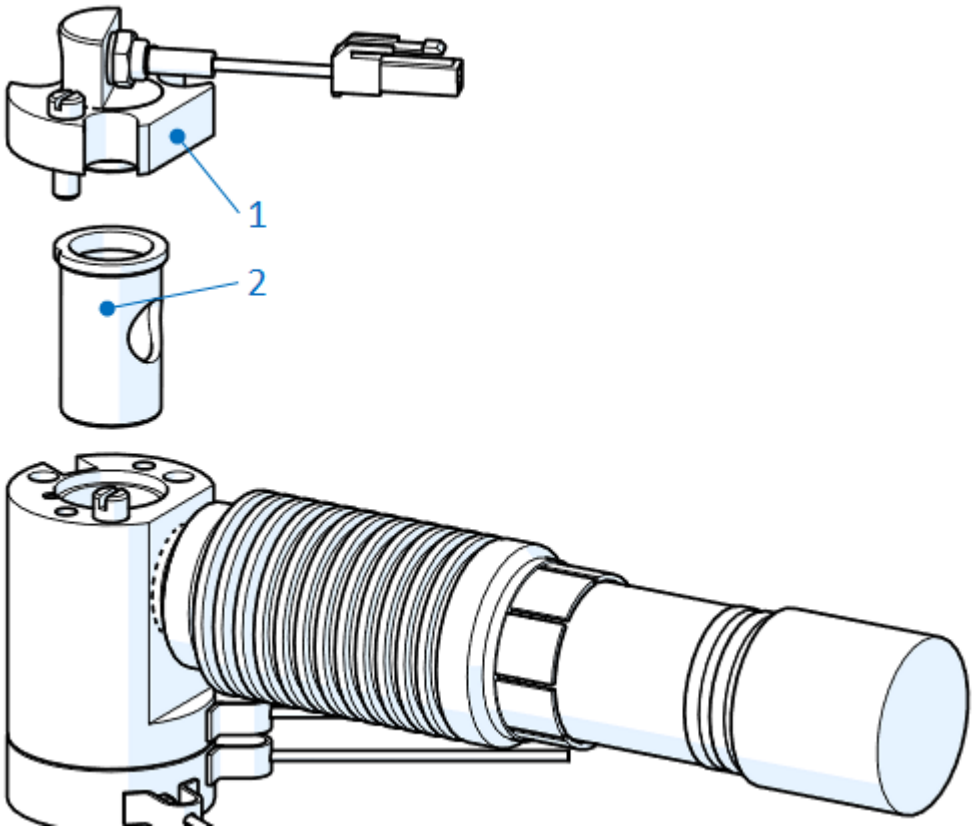
To eliminate this problem, it is necessary to carry out periodic cleaning detector working chamber and safety glass (quartz disk).

Procedures on FPD maintenance should be carried out at GC switched off.

Working chamber cleaning (Figure 3.49)

- Using screw-driver unscrew detector cover fasting screws (1);

- Remove the cap;
- Using pincers take out the replaceable chamber from the detector (2);
- Internal chamber surface is to be wiped thoroughly by rod with wadded tampon moistened with alcohol or acetone solvent.

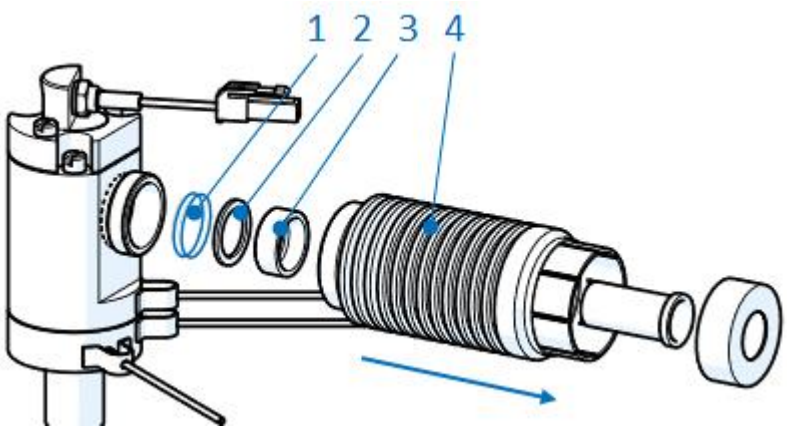


1 – Detector cover, 2 – chamber

Figure 3.14 –Chamber removing

Safety glass cleaning

- Using screw-driver weaken the PMT fastening screw;
- Take off PMT;
- Remove the cooling ventilator.
- Unscrew and take off PMT radiator (4);
- Using pincers take the pressure sleeve of protective quartz disk (1);
- Take out the rubber ring (2) and protective quartz disk by pincers;
- Clean both sides of quartz disk with the help of tampon moistened with acetone, alcohol or other solvent.



1 – quartz disk, 2 O-ring, 3 – pressing sleeve, 4 – PMT radiator

Figure 3.15 – Safety glass removing

At maintenance avoid touching quartz disk surfaces in order to prevent its pollution.

For better cleaning quartz disk, it is allowed to use "nitrohydrochloric acid" as a cleaning agent

3.6 Photoionization detector (PID) (P/N 150-2600)

3.6.1 PID operation principle

Photoionization detector (PID) is non-destructive detector of ionizing type. Its operation principle is based on photo-ionization of analytes by ultra-violet (UV) flux and consequent ions recording. Gas-discharge lamp filled with inert gas serves as UV radiation source. Current ions' recording is provided in ionization chamber.

Ionization in PID may be classified in 4 types. Direct ionization of registered analytes (AB) at photons influence (with energy $h\nu$) resulted in electron (e) emission and formation of molecular ion (AB^+):

$$\mathrm{AB} + \mathrm{h\nu} \rightarrow \mathrm{AB}^+ + \mathrm{e}$$

Ionization as a result of interaction between analytes molecules and carrier gas excited molecules C*:

$$\mathrm{C} + \mathrm{h\nu} \rightarrow \mathrm{C}^*$$

$$\mathrm{AB} + \mathrm{C}^* \rightarrow \mathrm{AB}^+ + \mathrm{e} + \mathrm{C}$$

Ionization by photons with intermediate transition of molecules AB in excited condition:

$$\mathrm{AB} + \mathrm{h\nu} \rightarrow \mathrm{AB}^*$$

$$\mathrm{AB}^* \rightarrow \mathrm{AB}^+ + \mathrm{e}$$

Side reactions responsible for charged particles recombination:

$$\mathrm{AB}^+ + \mathrm{e} \rightarrow \mathrm{AB}$$

$$\mathrm{C} + \mathrm{e} \rightarrow \mathrm{C}^-$$

$$\mathrm{AB}^+\mathrm{C}^- + \mathrm{AB} + \mathrm{C}$$

Table 3.9 – Ionization potentials of some substances

Component	Ionization potential, eV	Component	Ionization potential, eV
Permanent gases	>12	Acetone	9,7
Water	12,6	Ethanol	10,48
Alkanes C1–C4	10,6	Vinyl chloride	9,6
Carbon dioxide	12,8	Ethylene	10,5
Carbon oxide	14	Dimethyl sulfide	8,7
Benzene	8,2	Hydrogen sulfide	10,5
Toluene	8,8	Sulfur dioxide	12,3
Cyclohexene	8,9	Carbonyl sulphide	11,2
Cyclohexane	9,9	Ammonia	10,5
n-hexane	10,2	Nitrogen oxide	9,3

Molecules can be ionized if its ionization potential is lower than photons energy. Discharge lamp serves as a radiation source. Typically, krypton lamp with ionization potential 10,6 eV is applied with PID. By special order PID with argon lamp (ionization potential 11,2 eV) can be supplied.

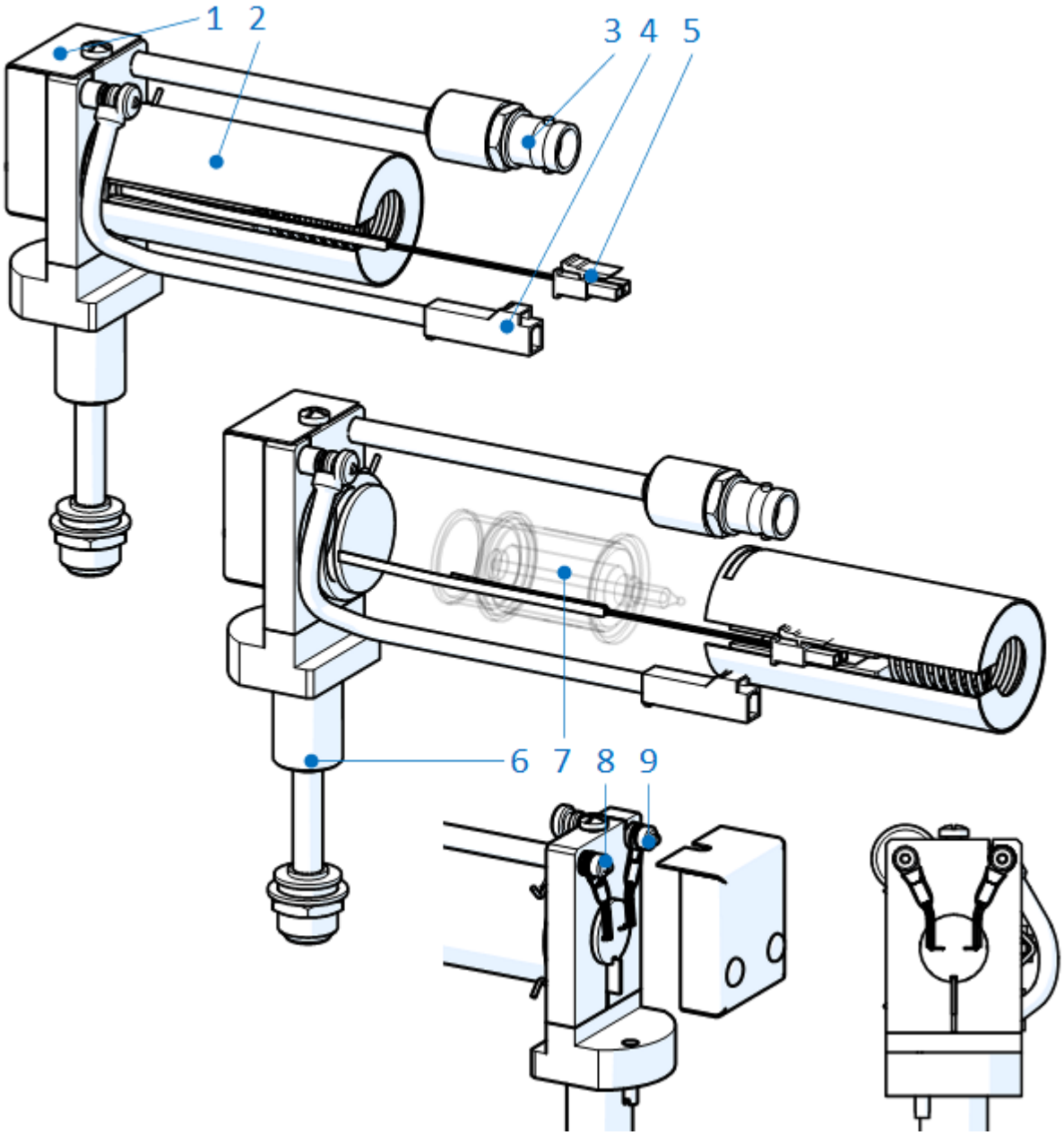
For standard substances and test conditions, the linear range of PID is over 10^7.

3.6.2 PID construction and installation

Detector (Figure 3.) contains the main elements: detector body 6, lamp 2 (ultra-violet radiation source), ionization chamber formed by isolator 3 and lamp window, polarizing and collector circuits.

Detector is installed into standard landing place of the heater. Detector input fitting passes through hole of heater and is tightened in oven by washer 14 and nut 15. Detector body is protected by cover fixed by 4 screws.

In ionization chamber electrodes are located: one of them (collector electrode) is connected to the amplifier and another one (polarizing electrode) connected to voltage source 200 V. Electrodes are connected to corresponding circuits by spring contacts (8, 9). To prevent mechanical damages, the lamp is protected by cover 5 which serves as electromagnetic screen as well. Ionization chamber and electrodes are protected by cover 1. The distance between polarizing and collector contacts (should make (1-2) mm.



1 – cover 8.634.474; 2 – cover 6.430.276 with spring 8.687.031 and isolator 7.890.006; 3 – contact 6.622.052 (to PID amplifier); 4 – cable 6.644.223 (polarizing voltage 200V); 5 – lamp power source (to 3mA power supply board); 6 –– detector body; 7 – lamp 5.132.055; collector; 8 – electrode contact 7.352.037; 9 – polarizing electrode contact 7.352.037

Figure 3.51 – PID detector 2.840.014-01.01

PID ionization chamber is made from teflon with maximal working temperature 250°C, therefore, in order to prevent detector breakdown, it is forbidden to set detector temperature above 250°C.

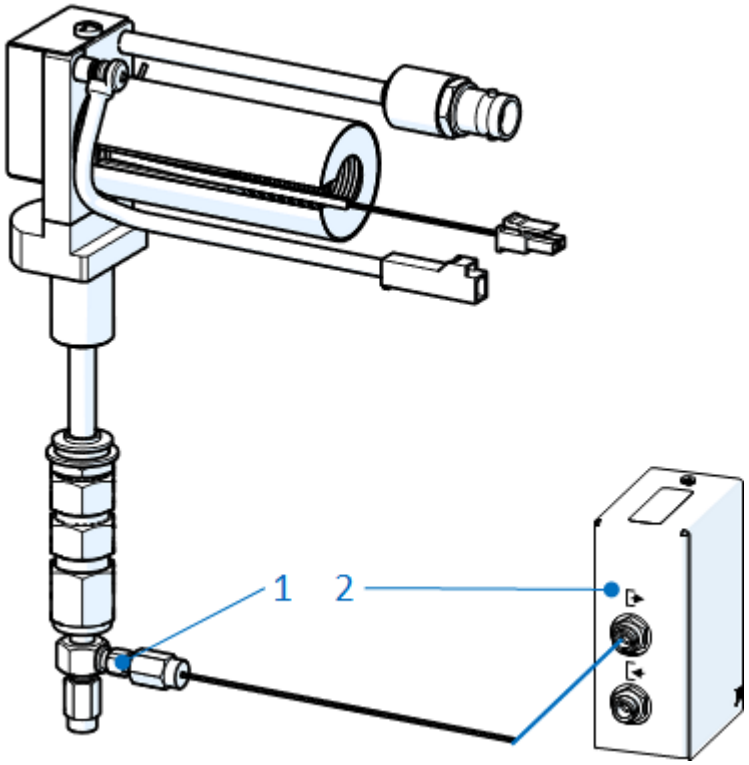
In PID high voltage for lamp supply is used. It is necessary to follow safety measures at detector installation.

At PID installation in the right-side compartment 2 boards are installed: 200V stabilizer, forming polarizing compartment, and 3mA stabilizer to supply lamp power.

Lamp is connected to D slot of 3mA stabilizer with the help of cable. When connecting, it's important to check polarity, for this purpose the point is marked on the lamp jack. 1-st pin of the cable corresponds to marked pin on lamp jack. For reliable connection cable jack is put on the detector guide.

3.6.3 PID pneumatic connections

Supply gases are not supplied to the detector itself, however, when using a capillary column, a blow-in gas with RRG-10 is additionally supplied to the tee 6.453.131-02 before the inlet fitting of the detector.



1 – T-joint 6.453.131-02, 2 – DFC-10

Figure 3.32 – PID gas connection for capillary column operation

3.6.4 Columns connection to PID

When attaching either capillary or packed column to PID, capillary 6.452.008–02 is used to transport gas flow from the column into ionization chamber. Top end of the capillary is adjusted at the distance of 2mm back from the chamber top wall. At greater gap, peaks tailing may occur. Capillary position can be checked visually, by taking out the lamp from detector.

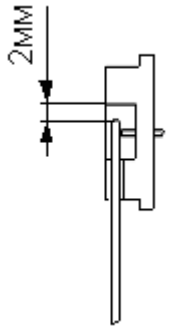
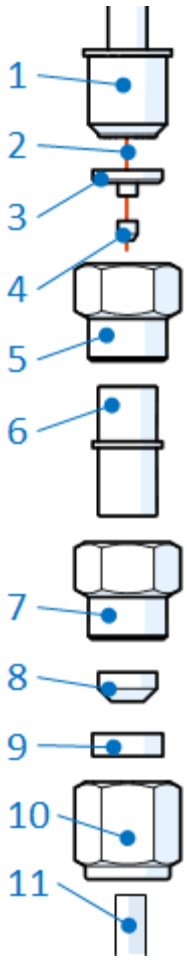


Figure 3.53 – Position of capillary in ionization chamber

Packed column ad capillary connection

- The installation of the capillary and fittings for working with the packed column is carried out in the following sequence.
- Fit guide 3 (8.224.073) and fitting 5 (8.652.307) on detector input fitting. Tighten the fitting finger tight. Use a wrench to tighten at additional 10-15 degrees.
- On capillary 2 (7.352.085), set ferrule 4 (cone down) as shown in the figure at a distance of 92 mm from the upper end of the capillary installed in the detector to the upper end of the ferrule.
- Install insert 6 (8.224.075) (cone-shaped hole for ferrule up) into the fitting 7 (8.652.307). Then carefully install the capillary with the ferrule cone down into the insert 6, maintaining the position of the ferule on the capillary.
- Insert the capillary with the entire assembly into the detector fitting. When installing and fixing the capillary, be careful to avoid its breakage. Screw the fitting 7 by hand, then turn by wrench for 10-15 degrees. At installation and fixing of capillary be careful in order to prevent its breakage.
- Insert the column end 11 with nut 10 and holder 8 (6.453.038) into fitting up to the stop. Tighten the nut finger tight. Use a wrench to tighten at additional 30-60 degrees

PID fitting for the packed column is lower than the usual fittings. Therefore, the second end of the column must also be installed using a special elongated fitting for the inlet (from PID detector shipping kit).



1 – Detector input fittig, 2 – capillary 214.7.352.085 (100mm), 3 – guide 214.8.224.073, 4 – ferrule 0,8 mm ID. № 072626 (SGE), 5 – fitting 214.8.652.307, 6 – insert 214.8.224.075, 7 – fitting 214.8.652.307, 8 – ferrule 214.8.220.380 (rubber), 9 – sleeve 214. 8.223.013, 10 – nut 214.8.930.172, 11 – packed column 5,4mm

Figure 3.16 – Packed column ad capillary connection

Capillary column connection

Before connection capillary column to PID detector, a capillary and the necessary elements 2-7 should be installed on the detector fitting, as shown above when installing packed column.

Capillary column is connected to PID detector via t-joint 6.453.131 03 (Figure 3.55). In the side fitting of the t-joint, make up gas is supplied into PID to compensate for the flow.

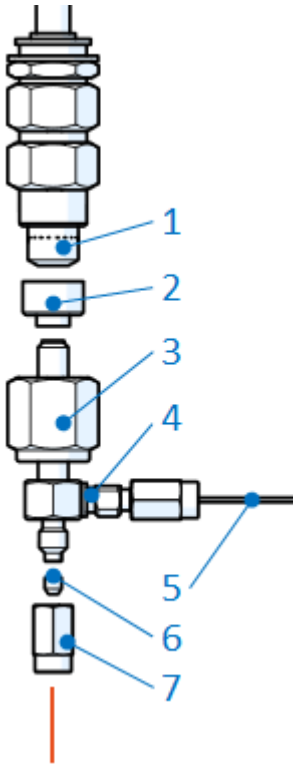
Nut 3 (8.930.172) and ferrule 2 (6.453.038) are installed sequentially on t-joint 4, as shown in the figure. The t-joint is inserted into the prepared adapter PID 1 until it stops, and is sealed with a nut 3. The tightening force of the nut is "by hand", followed by 180-270 degrees rotation for a new ferrule and 45-60 degrees for repeated tightening.

Side tube with a diameter of 1.6 mm is connected to FID make up gas line.

Put the nut 7 (8.930.339) and ferrule 6 (№ 072626 (SGE)) on end of capillary column.

Cut column end (5–20 mm). The cut should be flat without chips and scratches. Wipe cut end of the column with lint-free tissue wetted in alcohol in order to remove dust and other contaminants.

Insert the column upward to the stop and tighten nut is by hand. Then withdraw back at 1mm. Holding the t-joint 4 from turning with wrench (by 8), turn the nut 7 with the key (by 7) by 60-90 °so that column cannot be withdrawn from the fitting.



1 –adapter, 2 – ferrule 6.453.038, 3 – nut 8.930.172, 4 – T-joint 6.453.131-03, 5 – make up tubing, 6 – ferrule, 7 – nut 8.930.339

Figure 3.17 – Capillary column connection

If in work with PID the splitter into two detectors is used (for example, 214.5.885.013 or similar, providing required make up flow), then the piece of capillary column is installed into PID the same as with another detector (without T-branch fitting and capillary. The piece of capillary column thus does not reach the chamber top wall on 1 mm.

3.6.5 PID application features

Sensitivity

PID sensitivity decreases in the following lines of compounds:

- Aromatics > alkenes > alkanes.
- Ketone > aldehydes > ethers > alcohols.
- Cyclic > acyclic hydrocarbons.

PID sensitivity depends on molecule chemical construction: numbers of carbon atoms, nature and position of functional groups, double and conjugated double bonds.

At parallel detecting FID-PID the identification of complex samples by hydrocarbon classes can be provided with quite high authenticity.

PID response is proportional to concentration of analyte in ionization chamber and intensity of lamp ultra-violet radiation. Concentration of substance in chamber depends on carrier gas flow through it.

Carrier gas optimizing

The types of ionization, given in section 3.6.1, do not impose rigid restrictions on choice of carrier gas, as their ionization potentials are high enough. However, argon as a carrier gas is preferable as soon as electrons speed in its medium is higher, and its absorption of ultraviolet-radiation is lower, than in other gases. Accordingly, the response to benzene is reduced approximately in 1.5 times at replacement of argon by nitrogen or helium and in 5 times at replacement on CO_2.

Operation mode

Detector temperature. At high operation temperature of the detector sensitivity decreases and lamp lifetime shortens. Detector temperature is recommended to set at the minimum level (5-10 °C higher oven temperature), but not higher than 250 °C.

Optimal gas flow rate through detector (make up + flow through the column) is from 15 to 30 ml/min.

3.6.6 PID lamp usage

Lamp is the main component of the detector; its performance determines detector's sensitivity. Detector's sensitivity may reduce due to two reasons: pollution and limited lifetime of the lamp. Lamp cleaning procedure is described in section 3.6.7. Average lifetime of krypton lamp is 300-500 hours depending on detector temperature, detailed information is given in manufacturers accompanied documentation.

PM3 electronics has several settings to optimize lamp operation and extend its lifetime.

Initial condition of the lamp

In dialogue **Configuration** in Control panel of Chromatec Analytic SW choose command **PID / Lamp. Initial state**. On default the lamp is **Off**, detector signal is 0 mV. In this condition the lamp is off in any GC state (Not ready, Ready, Analysis etc.), if not intentionally turned on. Programmable lamp turning on during analysis is described below.

It's not recommended to use **on** initial position of the lamp while operation, this may be turned on in short-time to check lamp operability.

Lamp turning On/Off during Analysis

Period of time required to turn lamp on and ignite it for operation is obviously less than 5 sec, so lamp is recommended to be turned on while eluting target components only, this will extend lamp lifetime significantly. Other time the lamp is recommended to be turned off.

Lamp turning on/off setting is in dialogue GC instrument method in Control panel of Chromatec Analytic SW, option **PID/Switching**.

3.6.7 PID maintenance

While servicing PID, inevitable gradual pollution of ionization lamp window and detector chamber with sample and liquid phase components, consequence of that, can be essential decrease of detector sensitivity. To keep lamp at its highest performance, it's recommended to clean lamp window timely.



Contacts of lamp and chamber are under high voltage. All actions on assembly and disassembly of detector should be conducted only at the switched – off power supply of the GC.

Ionization lamp window cleaning

- Disconnect lamp power supply connector 200V;
- Remove detectors cover, weakening its fixing screw;
- Keeping lamp by fingers, remove holder fixing cover;
- Carefully remove cover together with lamp;
- Take the lamp out of the cover;



- Wipe lamp window with cotton applicator wetted with alcohol, until characteristic "glass" scratch appears;
- After cleaning lamp should be kept in the oven at temperature 100 °C for 10 minutes;
- Assemble the detector in reverse sequence.

When carrying out the above actions, do not touch the lamp window.

Do not immerse lamp window in the solvent entirely as this may damage lamp.

Sometimes, when lamp window is polluted, described procedure of lamp cleaning is not sufficient to restore lamp sensitivity. In this case cleaning by domestic light-abrasive cleaning agent (like Comet, Pemoxol) is recommended.

For cleaning follow instructions below.

- Take out lamp from the detector
- Wet lamp window and finger with tap water.
- Take on the finger-pad some cleaning agent (1-2 mm^3) and, touching lamp window slightly, wipe it in circular motion for 20…30 sec.
- Thoroughly wash cleaning agent out from the lamp with tap water.
- Wipe lamp window with cotton applicator wetted with alcohol, until characteristic "glass" scratch appears;
- After cleaning lamp should be kept in the oven at temperature 100 °C for 10 minutes.

At getting above mentioned procedure, some scratches may appear on lamp window surface. Frequent and intensive cleaning by light-abrasive cleaning agents may lead to window dimming and detector sensitivity loss. Therefore, this procedure is not recommended to carry out more often than once in 3 months. Do not press finger-pad tightly while cleaning.

Lamp replacement

At expiration of lamp lifetime, its sensitivity decreases gradually, lamp is subject to be replaced. Procedure on lamp replacement is the same as described above lamp taking out for cleaning purpose.

Once being replaced, calibration procedure is recommended.

3.7 Electron Capture Detector (ECD)

3.7.1 ECD operation principle

Operation of electron capture detector is based on capture free electrons by analytes molecules, free electrons are present in ionization chamber of detector.

Primary electrons, let out by radioactive source (beta-radiation), collide with molecules of carrier gas or make up gas (usually nitrogen). In result, secondary electrons and positive ions are formed:

$$\beta^* + N_2 \rightarrow \beta + N_2^+ + e^-$$

Between electrodes of detector there is weak electric field resulting in gathering of secondary electrons on anode and occurrence of initial current.

When analytes pass through ionization chamber of detector, the current decreases owing to capture of secondary electrons by analytes molecules according to reactions:

$$e^- + AB \rightarrow AB^- + h\nu$$

$$e^- + AB \rightarrow A^\bullet + B^-$$

In first reaction, negative molecular ion AB^- is formed, in second one molecules dissociation occurs, resulted in forming free radical $A^\bullet$ and negative ion B^-.

Chromatograph electronic scheme interferes with decrease of detector initial current, increasing electric field intensity between electrodes and supporting, by thus, constancy of this current rate. Change of electric field intensity, required for preservation of initial current invariance, is transformed to the detector output signal proportional to concentration of analyzed substance.

Rate of current proceeding through detector electrodes, corresponds to rate of basic current set from the chromatograph keyboard or computer.

For standard substances and test conditions, the linear range of ECD is over 10^4.

3.7.2 Using ECD with radioactive source (Ni63)

This section contains the information obligatory for Russian Federation. The regulation in other countries may be different. Please contact Chromatec local representative for details.

The detectors ECD (P/N 150-2501, Cross ID 2.840.040-01) and μ-ECD (P/N 150-2511, Cross ID 2.840.040-01.01) with the sources of ionizing radiation installed in them are radioisotope devices (RID).

An ECD detector 2.840.040-01 has a beta-radiation source radionuclide closed Nickel-63 source code BNi3.C3.4.R with an activity of 999 MBq.

In the μ-ECD detector 2.840.040-01.01, a beta radiation source with a radionuclide closed Nickel-63 source code BNi3.C10.1. R with an activity of 370 MBq is installed.

Radionuclide sources installed in the RID (ECD and μ-ECD detectors):

- According to potential radiation hazard, they belong to the 5th category (indicated in the passport to the source), which means – danger to humans is very unlikely (RB-042-07, NP-067-16, Appendix No. 2);
- According to the degree of radiation hazard, it belongs to group 2 according to SanPiN 2.6.1.3287 15, Appendix 1.

Activities using radiation sources containing only radionuclide sources of ionizing radiation of categories 4 and 5 of the radiation hazard are subject to registration in the manner established by the Government of the Russian Federation in accordance with Decree of the Government of the Russian Federation of 11/19/2012 N 1184 "On registration of organizations operating in the operation of radiation sources containing only radionuclide sources of the fourth and fifth categories of radiation hazard. "

Record and control is executed and fulfilled no later than 10 days after receipt (or dispatch) of the RID to the Regional Information and Analytical Center for Accounting and Control of Radioactive Substances and Radioactive Waste (hereinafter – RIAC) of the subject of the Russian Federation according to the form of a report in the field of state accounting and control of radioactive substances and radioactive waste according to the procedure and deadlines for reporting (Order No. 1/24-NPA of 09/28/2016). In the absence of RIAC, the report is submitted directly to TsIATS SGUK RV and RAO.

Installation, operation and disposal of RIDs should be carried out in accordance with the legislation of the Russian Federation: FZ-170 "On the Use of Atomic Energy", SanPiN 2.6.1.3287-15, NRB – 99/2009; OSPORB – 99/2010, SanPiN 2.6.1.1281-03.

Prior to the expiration of the designated service life of the ionizing radiation source (indicated in the passport for the ionizing radiation source), RIDs must be dismantled, and the sources contained in them must be disposed of in specialized organizations or returned to the RID manufacturer for subsequent disposal.

3.7.3 ECD (P/N 150-2501) construction

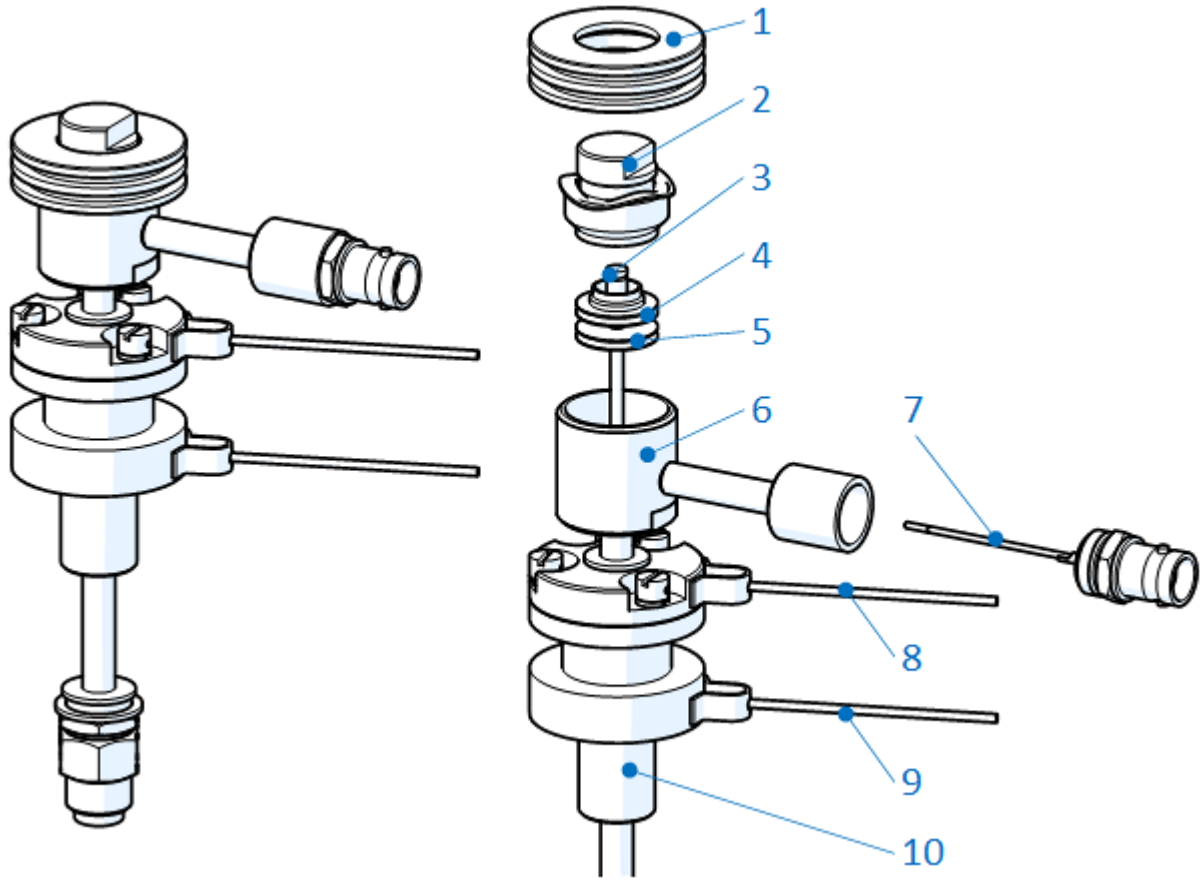
Detector ECD (P/N 150-2501, Cross ID 2.840.040-01) has bottom body 10 containing ionization chamber in which the radioactive source is installed. The source represents the nickel foil rolled as cylinder. On internal surface of cylinder, the layer of nickel – 63 covered with blanket of nickel is put. In ionization chamber the bottom part of electrode 3 serving as anode is placed. The cathode is the body 10 and radioactive source. Through the contact 7, electrode 3 connects to ECD amplifier. Parts 6 and 10 are pulled together with three screws with hinged seal.

ECD detector design allows the electrode to be removed for cleaning without the use of any tool. The extraction time of the collector electrode is no more than 1 minute.

The detector ECD (P/N 150-2501) contains the source of beta-radiation, radionuclide, closed Nickel – 63 (code BNi3.C3.4.R) with activity 999 MBk (27 mCi).

Do not disconnect parts 6 and 10. Do not tear hinged seal from the screws!

At high temperatures of detector, it is necessary to pay attention that detector top part can be heated to temperature, which can cause burn



1 – nut 8.936.016, 2 – cap (body 8.034.320) with spring, 3 – electrode (body 6.115.361), 4 – top insulator (washer 7.854.006), 5 – bottom insulator (washer 7.854.033), 6 – top body 6.113.322, 7 – contact 6.622.051, 8 – vent tubing, 9 – make up tubing, 10 – bottom body (6.115.150).

Figure 3.33 – ECD (P/N 150-2501) construction

3.7.4 µ-ECD (P/N 150-2511) construction

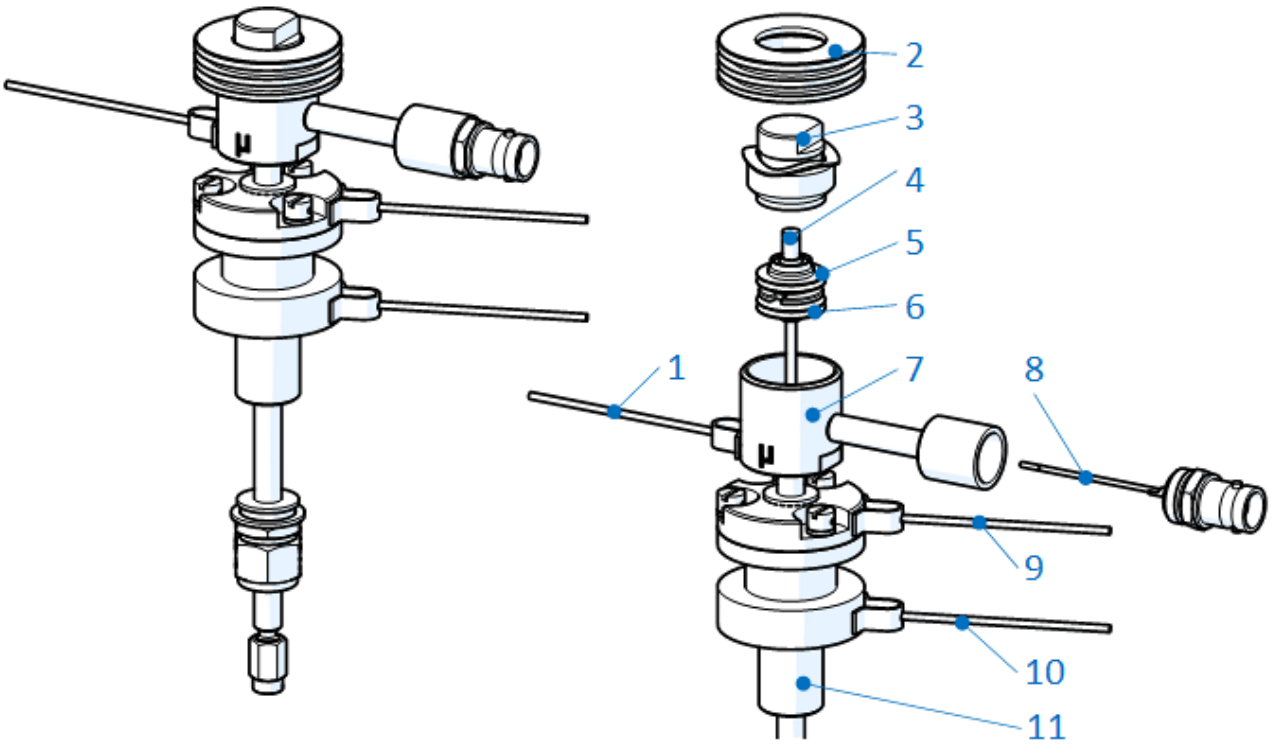
Detector µ-ECD (P/N 150-2511, Cross ID 2.840.040-01.01) is intended for operation with capillary columns, thus it has smaller ionization chamber volume than ECD (P/N 150-2501). In other features, µ-ECD (P/N 150-2511) design is similar to ECD (P/N 150-2501).

µ-ECD detector design allows the electrode to be removed for cleaning without the use of any tool. The extraction time of the collector electrode is no more than 1 minute.

The µ-ECD detector is designed to work with capillary columns and micropacked, it has a smaller working chamber and source, has a sensitivity of about 5 times higher than a conventional ECD detector. But the higher sensitivity made him more sensitive to pollution. To increase the resistance to pollution, it is necessary to reduce the concentration or volume of the injected sample, increase the division ratio of the sample in the inlet (if there is a margin of sensitivity) and apply a special operating mode.

Detector µ-ECD contains the source of beta-radiation, radionuclide, closed Nickel – 63 (code BNi3.C10.1.R) with activity 370 MBk (10 mCi).

At high temperatures of detector, it is necessary to pay attention that detector top part can be heated to temperature, which can cause burn

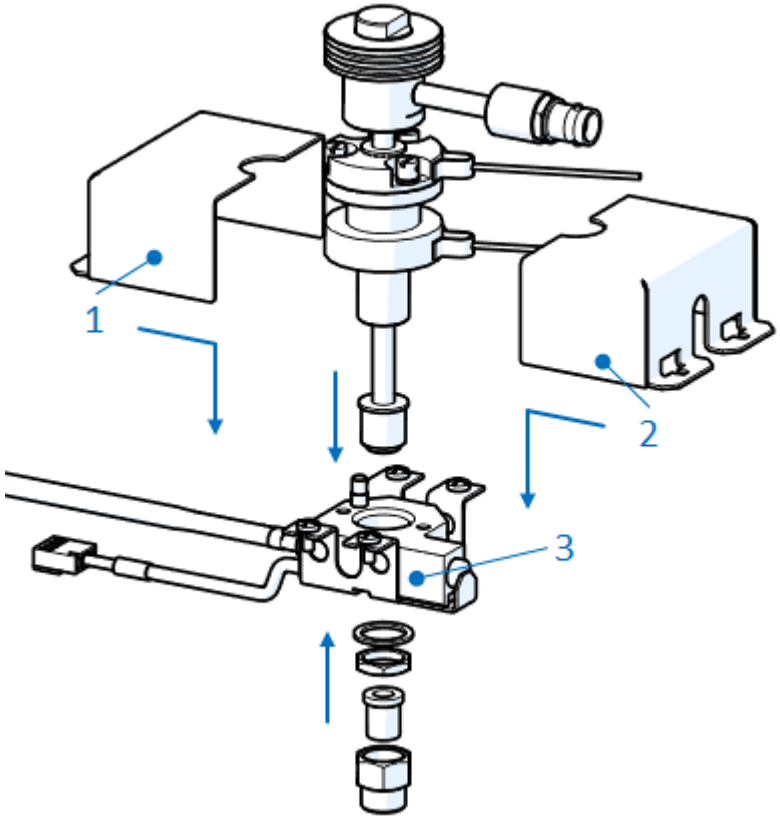


1 – anode gas tubing, 2 – nut 8.936.016, 3 – cap (body 8.034.320) with spring, 4 – electrode (body 6.115.283), 5 – top insulator (washer 7.854.006), 6 – bottom isolator (washer 7.854.033), 7 – top body (body 6.113.280), 8 – contact 6.622.051, 9 – vent tubing, 10 – make up gas tubing, 11 – bottom body 6.115.150.

Figure 3.34 – µ-ECD (P/N 150-2511) construction

3.7.5 ECD installation

Detectors ECD and µ-ECD are placed into standard landing place of the heater. Detector input fitting passes through hole of heater and is tightened in oven with nut. The pin in the detector body bottom part enters into heater centering hole. The detector bottom part is closed by covers 1 and 2



1,2 – Covers, 3 – heater

Figure 3.35 – ECD installation

3.7.6 ECD gas supply

Most frequently, for electron capture detector as make up gas is nitrogen. In some cases, argon with addition 5 vol.% methane is used. Argon/methane is recommended in case when wider linear detector response range is required, or electrons big mobility in ionization chamber is required at presence of pollution in carrier gas.

For carrier gas to ECD, Nitrogen or other gases, like Helium and Hydrogen may be applied.

Traces of organic admixtures, oxygen and waters vapors present in carrier gas and make up gas reduce free electrons concentration in ionization chamber, and consequently, and probability of their capture. Therefore, electron capture detector demands special cleanliness of gas lines and contents of oxygen and water vapors in carrier gas and make up gas less than 0.0001 vol.%. Using polluted carrier gas and make up gas may result in high level of detector signal, narrowing of detector dynamic and linear range and even distortion of useful signal.

Nitrogen 6.0 is recommended for the μ-ECD detector. When using a gas of worse purity, the detector's performance is not guaranteed.

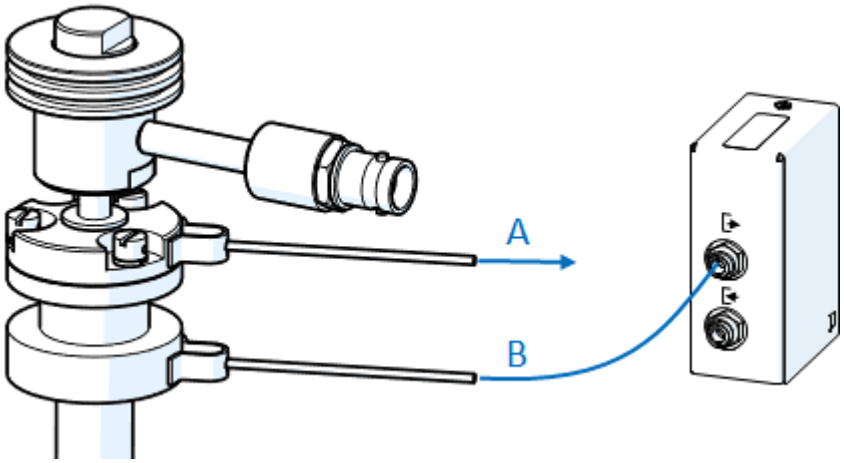
Chromatec Catalytic purifier 20.0 model 02 or 03 (P/N 451-0512, 451-0513) or universal filter trapping moisture, hydrocarbons and oxygen (e.g., P/N 1035348 Trajan) are recommended to purify carrier gas.

3.7.7 ECD pneumatic connections

Pneumatic feeding for ECD (P/N 150-2501)

Connection of "ECD make up" DFC10. Blowing in detector is carried out with the help of flow controller DFC10.

Anode gas line is plugged with Plug 8.632.206, nut 8.930.167-04 and ferrule 8.658.052.

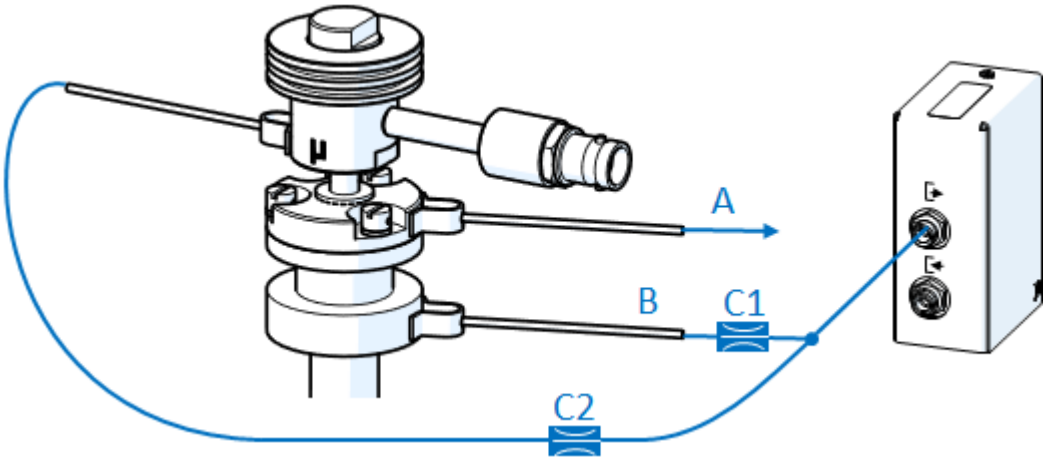


A – vent line, B – make up gas

Figure 3.18 – Pneumatic feeding for ECD (P/N 150-2501)

Pneumatic feeding for ECD (P/N 150-2511)

Make-up gas and anodic gas is supplied by DPC-11-02, the flow is equally divided in two flow restrictors. Flow controller on DPC11 shows the total flow rate of makeup gas and anode gas. To determine the flow rate of the makeup gas or anode gas, the readings of DPC11 must be divided into two.



A – vent line, B – make up gas, C1, C2 – restrictors, D – anode gas

Figure 3.19 – Pneumatic feeding for ECD (P/N 150-2511)

To prevent ambient air pollution by aggressive and toxic substances of analyzed sample, it's recommended to attach extended tubing to "ECD vent" line so that gases from ECD detector vent out of lab.

3.7.8 Columns connection to ECD

Packed and capillary columns can be connected to ECD. Only capillary columns can be connected to µ-ECD.

Packed column connection

It is recommended to use at a temperature in column oven less than 250 °C.

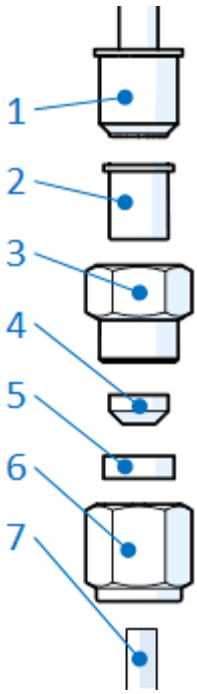
To connect packed column, install the insert 8.223.507 and the fitting 8.652.307. Tightening force of the fitting 8.652.307 "by hand" followed by turning the key by 10-15 °.

Put column end into insert 8.223.507 to the end. Tightening force of the fitting 8.930.172 "by hand".

Instead of ferrule 8.220.380 and sleeve 8.223.013, ferrule 8.453.038 (with graphite) can be used/

Graphite ferrules are allowed to use in the oven up to 450°C. Tightening force of the fitting 8.652.307 "by hand" followed by turning the key by 10-15 °. Place end of the column into insert up to the stop. Hold the column in this position and tighten the nut 8.930.172 finger tight. Use a wrench to tighten the nut at additional 30-60 degrees.

Be careful when tightening glass column with graphite ferrules in order to avoid its breakage.



1 – ECD fitting, 2 – insert 8.223-507, 3 – fitting 8.652.307, 4 – ferrule 8.220.380 (rubber), 5 – sleeve 8.223.013, 6 – nut 8.930.172, 7 – packed column

Figure 3.61 – Packed column connection with rubber ferrules

Capillary column connection

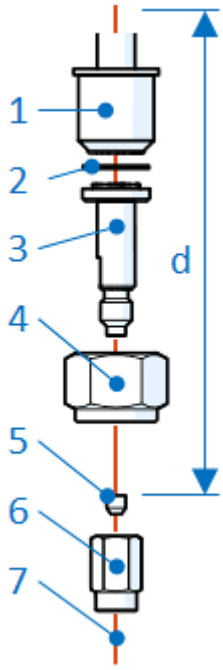
Fit adapter 6.115.318 before capillary column installation. Assembly of the adapter is shown on figure below.

Place washer 2 (8.942.247) on the insert 3 (6.278.008) and put it into nut 4 (8.930.268-01) as shown on the figure. Attach this assembly into fitting 1 of detector. Turn by hand, then tighten clockwise by wrench at 10-15°.

Put on nut 6 (8.930.339) and ferrule 5 (e.g., #072626 SGE, with its cone into nut) on the end of column 7.

Cut column end at (5–20 mm) to avoid graphite ingress into column. The cut should be flat without chips and scratches.

Wipe cut end of the column with lint-free tissue wetted in alcohol in order to remove dust and other contaminants.



1 – Detector fitting, 2 – washer 8.942.247, 3 – insert 6.278.008, 4 – nut 8.930.268-01, 5 – ferrule, 6 – nut 8.930.339, 7 – capillary column

d = 78 mm (for ECD)

Figure 3.62– – Installation of capillary column

Locate nut 6 and ferrule 5 at required distance (d = 78mm for ECD) from column end. For convenience, use column measuring tool 6.236.249 from shipping kit. Install column end into measuring tool, align column end at mark (3) for ECD detector and screw the nut so that column is fixed tightly by ferrule. Then release nut.

Insert the column into detector, tighten nut 6 by hand.

Then tighten nut 6 by wrench at 60-90 degrees so that column cannot be withdrawn from the fitting (column is tightened by ferrule holder).

3.7.9 ECD application features

Sensitivity and selectivity

Sensitivity and selectivity of detector is defined by affinity to electron (ability to capture electron) passing through substances ionization chamber, and also detector operating conditions. Strong affinity to electron, have molecules which have in its composition some atoms of halogens, for example.

For halogenated compounds, sensitivity of detector increases in line F <Cl <Br <I.

Relative sensitivity of the detector to some organic compounds is given below.

Table 3.10 – ECD relative sensitivity

Component	Relative sensitivity	Component	Relative sensitivity
Ethane	1	Chloroform	10^4–10^5
Benzene		Nitrobenzene	
Butanol	1–10^2	Carbon disulfide	
Acetone		Cinnaroic aldehyde	
Chlorobutane		Carbon tetrachloride	10^5–10^6
Chlorobenzene		Dinitrophenol	
1.2 dichlorethane	10^2–10^4	Diethyl fumarate	
Anthracene		Dinitrobenzene	
Ketosteroids		Hexachlorobenzene	
Tetraethyl lead		Hexachlorocyclohexane	
Benzyl chloride			

Linearity

The linear range of the ECD detector is not the same for the analytes. The detector characteristics give a linear range to lindane (γ-hexachlorocyclohexane). At the same time, for other substances the linear range can be only 2-3 orders. For this reason, when building a calibration curve for the response of the ECD detector, it is often recommended to use a point-to-point function

Recommended operating conditions

The higher detector temperature is, the better sensitivity of the detector to substances, capturing electrons with dissociation. Change of carrier gas or make up flow rate also have an influence on sensitivity as the response of detector depends on concentration of gas mix which passes through detector. Recommended ECD and μ-ECD operation mode is given below.

Recommended operating mode for ECD detector is given in Table 3.11. Recommended operating mode for μ-ECD detector is given in
Table 3.12.



Be careful to avoid injecting aggressive substances which can react with Nickel – 63, worsen quality of the Ni63 layer and form volatile compounds with Nickel – 63.

Table 3.11 – Recommended operation mode for ECD (P/N 150-2501).

Parameter	**Packed column**	**Capillary column**
Carrier gas	Nitrogen or Argon/Methane, (20–30) ml/min	Nitrogen, hydrogen or helium; flow (0.5–5) ml/min depending on column i.d.
Make up	Nitrogen or Argon/Methane, (20–30) ml/min	Nitrogen or Argon/Methane, (20–50) ml/min
Temperature	(250–400) °C. Temperature rate depends on analytes. Detector temperature to be set on 20 °C higher than maximum column temperature.	
ECD current	(100–150) rel. units	
Typical baseline level at manufacturing output	(200–300) mV	(80–150) mV

Table 3.12 – Recommended operation mode for μ-ECD (P/N 150-2511).

Parameter	**Mode for better sensitivity**	**Mode for high concentration (with better durability to pollutions)**
Carrier gas	Nitrogen 6.0, hydrogen or helium; flow rate (0.5–5) ml/min depending on column internal diameter	
Make up *	Nitrogen 6.0, (5-10) ml/min	Nitrogen 6.0, (15-30) ml/min
Anodic gas *	Nitrogen 6.0 or Argon/Methane, (5-10) ml/min	Nitrogen 6.0 or Argon/Methane, (5-30) ml/min
Temperature	(250–400) °C. Temperature rate depends on particular application. Set temperature value 50 °C above maximum column temperature	
μ-ECD current	20 rel. units	10 rel. units
Typical baseline level at manufacturing output	(60–200) mV	(70–250) mV

* Make up and anodic gas from one DPS 11 through two pneumatic resistances, the flow rate of makeup and anodic gas is calculated by dividing the flow rate readings of the DPC11 by two.

ECD current

ECD current is the reference current corresponding to rate of ionization current proceeding through detector. ECD current is set from the chromatograph keyboard or computer in relative units from 0 (0 nA) to 350 (4.1 nA). Set value depends on activity value of radioactive source installed in detector. ECD current is set at manufacturing. Standard value – 120, that corresponds 1.4 nA. In some cases, to improve signal/noise ratio, the current rate may be changed within ±50 units. Be aware, that at change of ECD current rate detector response is changed, therefore graduation and sample analyses should be carried out at the same current level.

Baseline signal level

Baseline level is output signal of detector at absence of analytes in the detector. Baseline level depends on purity of gases, chromatographic column bleeding, purity of detector itself, temperature of detector, gases flow rates, ECD current rate. Too high level of signal (500 mV and more) can testify that detector maintenance is required.

Maximum level of detector output signal for ECD is 10000 mV.

Maximum level of detector output signal for μ-ECD is 16000 mV.

Keeping a sufficient performance of ECD

The good performance of the ECD detector (sensitivity and signal level) during daily operation is a result of two possible factors:

- Good quality of feeding gases
- Clean detector chamber

If the detector chamber and / or supply gases become contaminated, the baseline signal of the detector inevitably increases, and the sensitivity decreases. The μ-ECD detector is more prone to contamination due to its higher sensitivity.

To maintain the required gas purity, it is recommended to use special filters, see section 3.7.6.

To keep the detector chamber clean, it is recommended that you follow a few simple rules:

- Set the operating temperature of the detector 50 ° above the maximum column temperature during analysis;
- To condition the column, disconnect the end of the column from the detector and plug the detector fitting in the oven;
- When analyzing dirty samples, use sample preparation methods that ensure the best cleaning of the sample before analysis (for example, solid-phase extraction in soil analysis cleans the sample better than liquid-liquid extraction).
- Adjust split ratio. Optimize the sample size injected into the column, just what is necessary to achieve sensitivity, and avoid extra sample from entering detector chamber.
- Run detector conditioning regularly.

3.7.10 ECD maintenance

The maintenance of the ECD detector is carried out periodically or unscheduled when the signal level is over 500 mV.

The signal of the ECD detector may increase due to inadequate quality of the supply gases or due to contamination of the detector and column. With increasing baseline signal, the sensitivity of the detector goes down. The purpose of maintenance procedures is to reduce the background to acceptable operating values (see section 3.7.9).

Table 3.13 – Procedures of ECD maintenance

Procedure	**Periodicity ***
Replacement of regeneration of the traps and purifiers in feeding gases lines	annually, or according to manufacturer's recommendations
Column conditioning	in 6 months, always prior to detector baking out
Baking out	in 6 months
Collector electrode cleaning	Always after baking out and cleaning detector
Cleaning the detector	by request, if baking doesn't help

* Whenever the signal of the detector is excessively increased, it is recommended to do maintenance.

Column or detector contamination is not a warranty case, and all maintenance procedures for cleaning can be carried out by the user.

Before conducting detector maintenance, it is necessary to find out the probable reason of the excessive signal level.

Purity of carrier gas and charge gas

Gas purifiers are not a part of ECD detector, but directly affect the operation and performance of the detector; therefore, when eliminating the causes of excessive signal level, it should be taken into account.

ECD detector is sensitive to oxygen impurities in carrier gas. If high baseline signal occurred right after gas cylinder had been replaced, then the reason of too high baseline can be gas from gas cylinder with the increased oxygen contents. Besides oxygen could get into GC gas supply system while gas cylinder replacement or after molecular sieve traps regeneration (if not had been thoroughly blown out before gas lines montage). To eliminate oxygen in this case additional time could require – up 3-4 hours.

Follow recommendations on gas cylinder replacement and purifiers installation for troubleshooting related to gas purity.

Column conditioning

ECD is non-destructive detector. That's why another reason for unsatisfied performance may be caused by the fact that heavy components exiting the column are deposited on the inner surfaces of the detector.

If detector baseline was increasing gradually during several days or weeks it is necessary to look for the reason of too high signal in column bleeding and pollution of detector with heavy sample components.

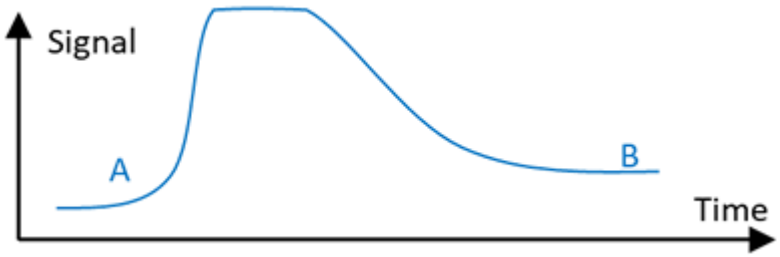
To clean column, it should be conditioned, see details in section 4.3 "Column conditioning" of part 1 User Manual. Column conditioning should always be done before baking out the detector.

ECD detector baking

Detector baking procedure takes from one to several hours, depending on contamination level. Baking procedure is described below.

Procedure:

- Disconnect and remove all columns from the oven; seal input detector fitting and inlet fitting in the oven;
- Set make up gas 100 ml/min;
- Set detector temperature 400 °C, oven temperature 350 °C;
- Start detector signal record (command **Start data acquisition** in "Chromatec Analytic" SW). While temperature increasing, signal grows rapidly (even up to maximum value), then goes down gradually. Typical detector signal record at baking looks like:



A – signal grows start, B – signal stabilization

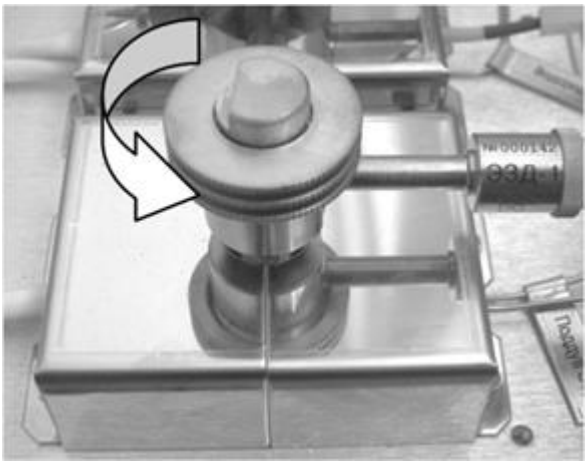
Figure 3.20 – Signal change during detector conditioning

- As soon as signal level stabilizes, oven and detector temperatures may be decreased to operating mode. Columns are to be installed in the oven.

After baking procedure, conduct cleaning of the electrode (see below).

Electrode cleaning

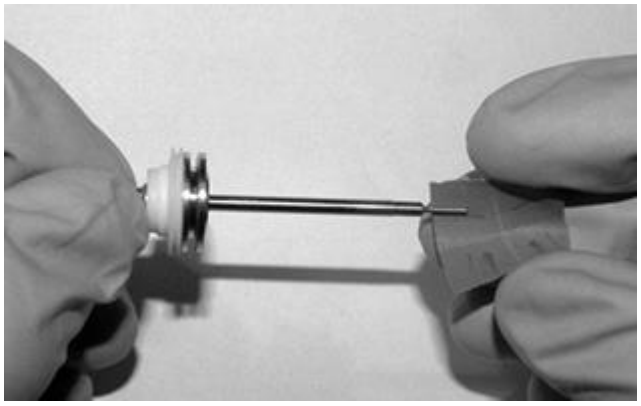
- Remove detector top part, unscrewing the nut;
- Disconnect amplifier jack and unscrew contact;



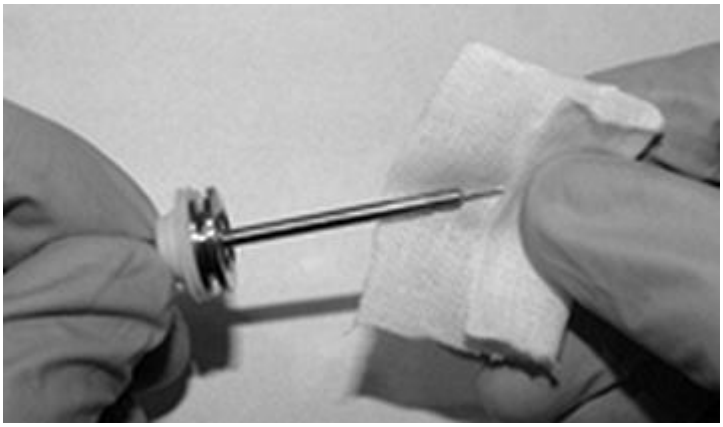
- Further procedures (hands should be in gloves):
- remove electrode and isolators;



- Sand tip of electrode with thin abrasive paper (#1000 or higher);



- Wipe electrode surface with lint-free tissue with acetone;



- Assemble the detector in reverse sequence.

Sometimes, after cleaning the electrode, the baseline signal of the detector can drop to 0 V. To bring the detector out of this state, it is advised to increase the temperature of the detector to 400 ° C for 1 hour, and then lower it to the operating value.

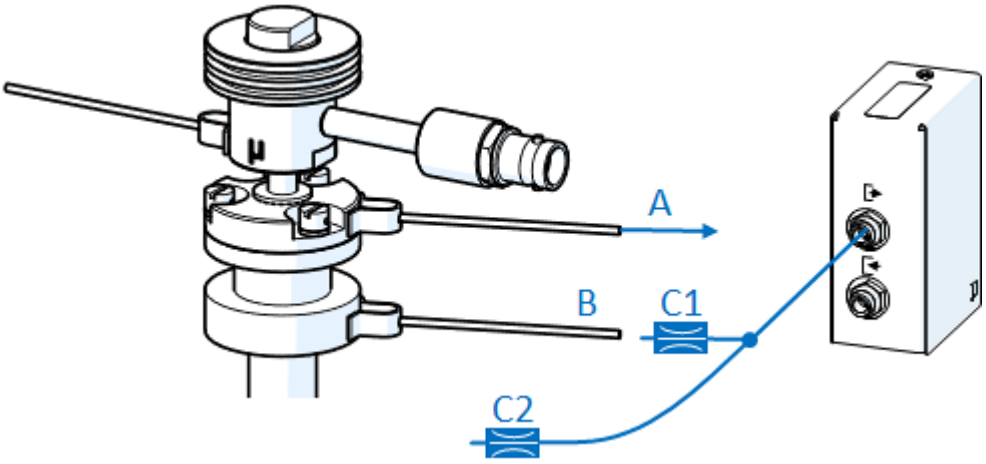
Cleaning the detector

The procedure for cleaning the detector is carried out only if baking procedure and cleaning the electrode did not give a positive result.

In accordance with the requirements of sanitary rules and regulations for the installation and operation of radioisotope devices SanPiN 2.6.1.3287-15 "Sanitary and epidemiological requirements for the handling of radioisotope devices and their device" the body of the electron-capture detector is sealed. Therefore, of all parts of the detector, only the electrode and its insulators are accessible for maintenance.

Detector cleaning consists in intensive cleaning of the internal channels and detector chamber with solvent vapors.

- Disconnect and remove the columns from the oven; plug the fittings of the detector and the inlet;
- Remove the upper part of the detector by unscrewing the nut.
- Disconnect make up and anodic gas lines of the detector from the restrictors or DFPC.



- Set the temperature of the detector and the oven at 350 ° C, you can skip setting gas flows;
- In 10 minutes, upon reaching the set temperatures, it is necessary to open the door of the column thermostat (**OBLIGATORY!**)
- Take 3-5 ml of ethyl alcohol into a syringe slowly introduce it into the make-up line of the detector. Instead of ethyl alcohol, you can use hexane, or a mixture of Hexane: Acetone (1:1). Different solvents have different efficiency in flushing depending on the nature of the contaminants.
- Put the chromatograph in cooling mode and, after cooling the chromatograph, assemble the detector, restore its gas supply and install a column.
- After cleaning the detector, it is always necessary to clean the electrode (as described above) and warm up the detector at 400 ° C for 1 hour.
- Install the column and connect it to the inlet and the input detector fitting.

3.8 Mass spectrometric detector (MSD)

SDO Chromatec provides the following documents for the MSD Chromatec:

- Mass spectrometer detector. User manual 214.2.840.083UM.
- Mass spectrometer detector. Passport 214.2.840.083PS (EI option) or 214.2.840.083-01PS (EI + CI option).
- MSD Direct probe system. User Manual 214.5.880.003UM.

The software for the MSD Chromatec is Chromatec Analytics, ver. 3.1.

3.9 Pulsed discharged detector (PDD)

At operation with PDD follow the documents:

Pulsed Discharge Detector Models D-2 and D-2-I Instruction Manual (Valco Instruments Co. Inc.), particularly D-2 version (multipurpose detector).

3.10 Helium ionization detector (HID)

At operation with PDD follow the documents:

Pulsed Discharge Detector Models D-2 and D-2-I Instruction Manual (Valco Instruments Co. Inc.), particularly D-2-I (HID optimized version).